

This has brought about the idea of oil manufacturers employing practical and experienced men educated in actual service to follow up and watch the proper application and economic use of the various lubricating oils and greases. These men have demonstrated, not only to their employers, but also to the consumer, the practical and economic value of their educational work.

TABLE 5.—CONSTANT-TEMPERATURE TESTS, GALENA OIL TESTING MACHINE.

Comparative frictional tests between pure rape-seed oil and winter Galena railroad car oil (zero cold test oil).
Steel journal, size 5" X 9"; bearing, genuine babbitt; total load on bearing, 10,000 lbs.; projected area, 15.5 sq. in.
Area of contact, 16.40 sq. in.
Pressure per sq. in. projected area, 645 lbs.
Manner of lubrication, oil-bath.
300 revolutions per min. = 392.5 ft. surface speed, average friction of four tests for each temperature.

Rape-seed oil.		Winter Galena car oil.	
Total fric. in lbs.	Coef. of friction.	Total fric. in lbs.	Coef. of friction.
50° F. 21.06	0.00211	50° F. 20.37	0.00205
70° F. 15.375	0.00154	70° F. 14.54	0.00146
90° F. 13.875	0.00139	90° F. 12.75	0.00128

600 revolutions per min. = 785 ft. surface speed, average friction of four tests for each temperature.

Rape-seed oil.		Winter Galena car oil.	
Total fric. in lbs.	Coef. of friction.	Total fric. in lbs.	Coef. of friction.
56° F. 26.25	0.00263	60° F. 19.94	0.00199
70° F. 25.375	0.00254	70° F. 18.31	0.00183
90° F. 19.75	0.00198	90° F. 15.31	0.00153

Viscosity (P. R. R.):

Rape-seed oil.	Winter Galena car oil.
At 125° F. 80 units	72 units
100° F. 125 "	104 "
90° F. 141 "	125 "
80° F. 186 "	160 "
Cold test +15° F.	-5° F.

By constant temperature is meant that the oil-bath and bearing is kept at uniform constant temperature during the whole time of test, which lasts not less than one hour after the desired constant temperature of oil and bearing is reached.

The importance of this "following up" is far-reaching. It has gradually brought about much more systematic and uniform method or methods in lubrication, it has brought about greater economy in the consumption of lubricating oils, and at the same time demonstrated better lubrication; in fact, in many instances brought down the consumption of oil from 50 to 100 per cent., without impairment of the best and most economic lubrication. This, of course, means a saving of large sums of money by the consumers, and satisfaction to the company in the knowledge of giving its patrons the best possible service.

From these remarks, you will readily appreciate that to make laboratory tests of lubricants of real practical value, not only to the consumer but also to the manufacturer, involves considerable technical and practical knowledge and experience, besides full and complete laboratory equipment, and the chemist or engineer who is called upon to give a qualified opinion as to the relative, comparative lubricating values of two oils or greases for a given service, considered from

a practical and economical service standpoint, has indeed a difficult and oftentimes thankless task to perform.

TABLE 6.—CONSTANT-TEMPERATURE TESTS, GALENA OIL TESTING MACHINE.

Steel journal, 5" X 9"; bronze bearing; 7800 lbs. total load on bearing; 300 lbs. pressure per sq. in.; 27.7 sq. in. area of contact; 363 rev. per min. = 475 feet surface speed; manner of lubrication, oil-bath; average friction of four tests for each temperature.

Temp. 65° F.	Galena winter car oil, lbs.		Galena summer car oil, lbs.	
	Friction, right.....	18.50	18.50	41.00
Friction, left.....	18.50	18.50	41.50	41.50
Friction, average.....	18.50		41.25	
Coef. of fric.....	0.00237		0.00529	
Mean resistance per sq. in. of surface.....	0.665 lb.		1.489 lbs.	
Temp. 80° F.				
Friction, right.....	15.50	15.00	29.50	28.50
Friction, left.....	15.00	15.00	30.25	29.50
Friction, average.....	15.125		29.44	
Coef. of fric.....	0.00196		0.00377	
Mean resistance per sq. in. of surface.....	0.546 lb.		1.063 lbs.	
Temp. 100° F.				
Friction, right.....	11.25	11.00	20.00	20.00
Friction, left.....	10.00	10.00	20.00	20.00
Friction, average.....	10.563		20.063	
Coef. of fric.....	0.00135		0.00257	
Mean resistance per sq. in. of surface.....	0.382 lb.		0.724 lb.	

The mean resistance per square inch of surface obtained by dividing the average total friction by the number of square inches (27.7) area of contact.

	Galena winter car.	Galena summer car.
Flashing point.....	315° F.	395° F.
Burning point.....	370° F.	455° F.
Gravity at 60° F.....	27.3° Bé.	24.3° Bé.
Cold test.....	+2° F.	+36° F.
Viscosity (P. R. R.):		
Pipette, at 125° F.	83 units	189 units
100° F.	124 "	343 "
90° F.	156 "	473 "
80° F.	201 "	...

METHODS OF ASPHALT EXAMINATION.

By ALBERT SOMMER.

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The present chemical knowledge of bituminous substances is undoubtedly most limited, and their practical chemistry is confined to the most rudimentary tests, basing on "rule of thumb" methods rather than on an exact system of chemical analysis. The lack of such scientific analytical methods has always been felt more or less keenly, but never more than at present.

The commercial importance of bituminous products has been vastly increased by the application of those materials to country roads, and the following discourse deals largely with asphaltic materials applicable for that purpose.

Bitumen available to-day for this purpose originates largely from the following sources:

A. *Residuals.*

1. So-called "native" bitumen, such as deposits of Trinidad, Bermudez and similar ones.
2. Residues from crude petroleum oil. These may be divided into:
 - (a) Residue from pure asphaltic oils.
 - (b) Those from semi-asphaltic oils.
 - (c) And, also sometimes those from straight paraffine oils.

B. *Distillates or Pyro Products*

3. Coal gas tar.
4. Water gas tar.

Inasmuch as all "natural" asphalts in class 1 undoubtedly represent residuum of petroleum, evaporated by natural causes, there is no strict dividing line between those products and the ones originating from asphaltic petroleum, reduced artificially in stills; and there being no essential, chemically differentiating features, any qualitative discrimination is unwarranted.

Furthermore, in order to become applicable, these natural products have to be softened by "fluxing," or the addition of oil residuum (especially in the case of softer road asphalts) which latter then more and more predominates and transfers into the compound its own characteristics.

Tar, however, and its derivatives are distillates or pyro-bitumen and naturally totally different from the previous two divisions.

The following is confined to a short description of methods either entirely new, or recently applied to asphaltic materials.

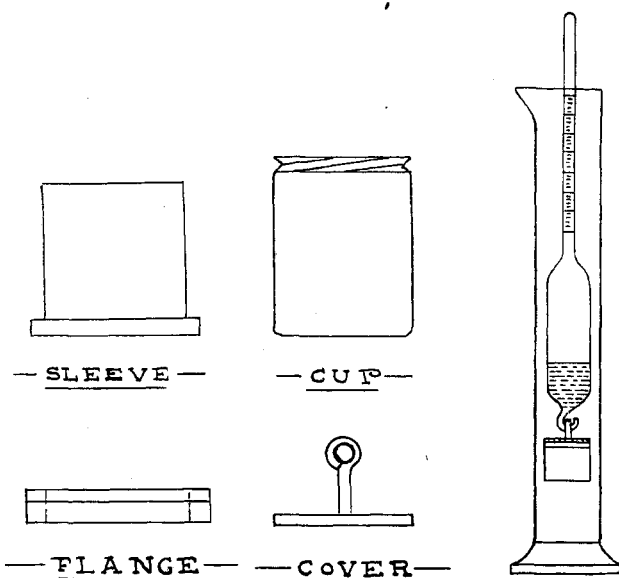
1. *Specific Gravity.*—It is remarkable how little attention has been paid to this part of bituminous research, whereas specific gravity is always one of the first means of identification of oils and other liquids.

Naturally the difficulties in determining the gravity of asphalts were considerable, inasmuch as by means of the hydrometer the gravity has to be taken at a temperature far above the ordinary one, and again, to take the gravity in a common pycnometer is also connected with the greatest difficulties on account of the shrinkage of bituminous materials when solidifying. An apparatus by which it is possible to determine the specific gravity of bitumen, in a quick and accurate way, in its normal consistency, was described by the author in a paper read before the American Society for Testing Materials, in July, this year. The principle of the method consists in letting the asphalt chill in a vessel consisting of two parts, the upper part of which takes up the shrinkage and is removed after the asphalt is chilled to a normal temperature. The lower part contains exactly 10 cc. and the gravity can be ascertained either by weighing out directly or by suspending the lower cup from a special hydrometer in water of 15.5° C.

2. *Determination of Asphalt.*—To one not familiar with commercial conditions it may appear peculiar upon first sight to hear of "percentage of asphalt" where asphalt has not as yet been chemically defined, and cannot, therefore, be determined like other chemical individuals. Nevertheless, in many instances it is essential to give a determination which is commercially satisfactory; hence a number of methods have been devised which are necessarily more or less arbitrary. The importance of determining asphaltic contents has become more appreciated since the introduction of semi-liquid asphaltic substances in the building of roads, for making so-called "bituminous macadam." It may be stated that the only reason for applying bitumen of such consistency is the necessity of incorporating it with cold stone under ordinary conditions; hard asphalt (such as is used in sheet asphalt pavement) would chill in contact with cold stone, and is therefore excluded, though it would otherwise be chosen, inasmuch as a certain hardness is required to make a satisfactory and permanent binder. For this reason such "cold" macadam binders must combine the highest percentage of hard asphalt with the highest possible fluidity, in order to be applicable; therefore the determination of asphaltic content in the same is of utmost importance to the purchaser of such materials. Unfortunately the difficulties are great and it can by no means be stated that they have at this date been solved. The present means of ascertaining the percentage of asphalt in such compounds are the following:

- (a) Precipitation with petroleum ether.
- (b) Precipitation with alcohol-ether.
- (c) In case of soft materials, evaporation until a certain consistency is reached.

It might be said that methods "a" and "b" tend to determine the percentage of asphalt by a chemical



Apparatus for determination of specific gravity.

reaction, precipitating a part of the same, while method "c" would define this percentage by reducing the bitumen to a certain, fixed, physical consistency, pronouncing anything "asphalt" which has that consistency or is harder. As to the first method with petroleum ether, it is known that petroleum ether precipitates the so-called "asphaltenes," whereas it dissolves the so-called "petrolenes." The difference between the two, however, is not sharp, as they are by no means defined chemical individuals, and their ratio is largely dependent on the solvent power of the petroleum ether used. Usually this ether is specified by gravity alone; for instance 86° Beaumé (= 0.6480 specific gravity) but it has been shown by numerous investigations by Holde, and also in the writer's laboratory, that petroleum ethers distilled from different crudes have different solvent power and that, therefore, beside the gravity, a distillation test should also be specified as lately recommended by Holde, the combination of both insuring absolute uniformity. Holde calls a petroleum ether of definite specific gravity and distillation "normal benzine."¹ In our laboratory we apply a distillate of 86° Beaumé gravity, distilling between 100 and 240° F. This combination of gravity and distillation tests insures a Pennsylvania product and therefore absolute uniformity in the results of precipitation. The precipitation by petroleum ether is of course not a direct measure for the real contents of asphalt, as it permits only of an approximate estimation of the same by the percentage of asphaltenes, provided that the nature of the bitumen is previously known; and furthermore, if the asphaltic material contains some light distillates, these very frequently act as a solvent and hence the precipitation with 86° naphtha shows less asphaltenes than it would if those light oils were not present. In the writer's laboratory this precipitation is performed by the following method:

Two grams of asphalt are weighed into a 100 cc. Jena glass flask, covered with 75 cc. of the normal petroleum ether mentioned above, and shaken until the entire asphalt is disintegrated. The flask is then allowed to stand over night, after which the solution is decanted and the precipitate poured into a small filter. An additional quantity of petroleum ether is then poured into the flask, rinsed and also poured through the filter, this being repeated with additional quantities of petroleum ether until the same runs off from the filter clear in color. After this a quantity of benzene is poured into the flask, which will dissolve all precipitate which may stick to the glass; then a weighted china dish is placed under the filter and the same benzene poured through the filter, the solution being received in the china dish. Benzene is applied in small quantities until all the asphalt

¹ Normal petroleum ether might be preferable to normal benzine on account of the similarity between "benzine and benzene."—Ed.

precipitated in the bottle, as well as on the filter, is dissolved and contained in the china dish. The benzene is then evaporated on the water bath until the dish has attained constant weight.

Should the asphalt contain anything non-soluble in CS₂, this of course will have to be deducted from the asphaltenes before figuring their percentage.

Precipitation by Ether-alcohol.—This method is not much in use in this country, although it combines a good many advantages, as will be shown further below by experimental data. Ether-alcohol precipitates not only asphaltenes but also the softer components of asphalt, and is therefore used especially to determine the total amount of asphaltic matter in crude oils, etc. The tables below will indicate that the method can also be well adopted for the determination in fluxes and harder bitumen.

The procedure for such grades is as follows:

Two grams of the asphalt are weighed into a 100 cc. graduated cylinder with a well-fitting glass stopper, and 40 cc. of a mixture of alcohol-ether, consisting of four parts alcohol and three parts ether, poured in the same cylinder. The same should then be attached to a shaking device and shaken well for about twenty minutes. After this time it should be left to stand over night, and should be filtered in the same manner as described with petroleum ether, only the same mixture of alcohol-ether should be used until all the soluble parts are removed from the precipitate. Benzene is then used in the same manner as above described, and the weighing and calculation made in the same manner as with the asphaltenes.¹

Determination of Asphaltic Contents by Evaporation.—This is the one most generally used in this country, especially on softer bituminous products. A certain quantity of asphalt is evaporated in a dish of certain dimensions, at a certain temperature. The loss is taken and consistency of the residuum determined by the usual methods such as penetration, *i. e.*, the number of tenths of mm. which a needle, weighted with 100 grams, penetrates in the asphalt at 25° C. Ten mm. of "100 penetration" is usually the chosen standard, and the percentage of remainder of that consistency usually called "asphaltic contents." Unfortunately the percentage thus found is not always the real one, as will be shown in the following:

Regardless of origin, there are two classes of such binders on the market at present.

1. Semi-liquid oil residuum, which is drawn from the stills of a consistency which makes it applicable to cold stone because of its physical characteristics alone.

Natural and lake asphalts such as Trinidad and Bermudez, fluxed with oil residuum to consistency, would of course also belong to this class.

¹ In the case of oils which are very rich in paraffine this method sometimes precipitates part of the latter also. Therefore the precipitate of such oils should be repeatedly extracted with hot alcohol.

2. So-called "cut back" materials which are made from a hard but pliable asphalt by the addition of enough light distillate to render the compound applicable, with the view of having the liquid serve only as a thinner or vehicle, much the same in principle as thinning varnish.

TABLE 1.—PERCENTAGES OF ASPHALT BY VARIOUS METHODS.

	Compound "A" (asphalt base).	Residual flux "B" (asphalt base).	Residual flux "C" (semi- paraffine).	Residual flux "D" (semi- paraffine).
1. Gravity, specific.....	0.986	0.980	0.963	0.980
Beaumé.....	12	12.9	15.4	12.9
2. Viscosity, Engler, at 100° C., 50 cc. run.....	195 sec.	150 sec.	62 sec.	165 sec.
3. Asphalt by 86° naphtha	14.72%	4.56%	2.30%	4.2%
4. Asphalt by alcohol ether.....	35.40%	17.20%	18.10%	41.3%
5. Asphalt by evaporation: (a) Per cent. of 100 pene- tration residue.....	88%	80%	68%	88%
(b) Time of evaporation	1½ hrs.	10 hrs.	12¾ hrs.	10 hrs.
6. Engler distillation: ¹				
16% distilled off, boil'g	170° C.	205° C.	295° C.	266° C.
5% off at.....	200° C.	325° C.	350° C.	350° C.
10% off at.....	260° C.	345° C.	360° C.	360° C.
16% off at.....	340° C.	361° C.	360° C.	370° C.
Gravity of distillate....	38.3° Bé.	29.1° Bé.	33.2° Bé.	34.1° Bé.
Viscosity in pipette....	11 sec.	21 sec.	18 sec.	17.0 sec.
7. Paraffine, Holde method	0.78%	0.29%	3.72%	3.35%
Melting point.....	138° F.	130° F.	121° F.	134° F.

From the standpoint of an oil refiner naturally the easiest way is to put out a product of class No. 1; whether, however, this is best from the standpoint of usefulness or efficiency is another question. This difference has been rather overlooked in the past and the results shown in Table 1 should serve as an illustration of the difference in various materials. Three types of fluxes were chosen for this investigation:

1. Compounded as above described (marked "A").
2. A residual flux (marked "B"), originated from an asphalt base petroleum as per Class No. 1, and
3. Residual fluxes (marked "C" and "D"), obtained from semi-paraffine base crude oils also per Class No. 1.

All semi-liquid asphalts used for these tests represent types of commercially available products,² such as used in actual work, and, all had nearly the same consistency, namely, semi-liquid. Table 1 shows percentages of asphalt of all these binders, as obtained by the various methods. The results of this table show that there are marked differences in true asphaltic content, which are to an extent illustrated by the results of precipitation with petroleum ether and also with alcohol-ether. The results of evaporation also show differences, but the mere percentages thus obtained do not illustrate the true nature of the various fluxes and, what is more important, do not show whether the asphalt which was thus found was already present in the flux or had been formed during the evaporation test itself. It cannot be emphasized too strongly that in all evaporation tests this will

¹ Note: Evaporation taken on 100 grams, in 5" × 2 3/8" box, at 500° F., in the open without stirring.

² And all marketed as "90 per cent. binder."

have to be reckoned with, although very few specifications at the present time take this into consideration. One glance at the table shows that the time required for the various evaporations indicates vast differences, and this already proves that percentages of asphalt obtained by the evaporation method in an unlimited time and high temperatures are often "pseudo percentages" which are of no practical value, inasmuch as such distilling or baking will never take place when those materials are in actual use. Practical experience has in the meantime confirmed these differences.

Precipitation and evaporation combined show that the hard asphalt obtained from residual fluxes was not originally present in the same, but has been produced by distilling or "cooking" heavy oils. In other words, their consistency is due to the presence of some asphaltogenous substances, which, while not being asphalts themselves, represent, as may be said, asphalt in an embryonic state which will form asphalt by the influence of great heat and oxygen; and combined with heavy, viscous, residual oils which can only be driven off at high temperatures; whereas the fluidity of "A" is caused by the admixing of a liquid vehicle to an already present base of hard asphalt, very much like the process of making varnish out of gum and turpentine.

The writer applies a method of distinguishing those products successfully by the determination of the consistency of the distillate obtained from such compounds. The distillation is performed in an ordinary Engler apparatus. We distil off a certain percentage of the compound, noting the temperature and if the same suddenly rises or jumps this in most cases indicates presence of a light thinner, whereas a residual oil will distil off more evenly; at any rate, to distil off about 20 per cent. will be sufficient in most cases. Distillates are then compared by gravity and viscosity and results shown in Table 1 exhibit the differences which may thus be obtained.

TABLE 2.—VARIOUS RESULTS OF EVAPORATIONS UNDER DIFFERENT CONDITIONS.

	Compound "A" (asphalt base).	Residual flux "B" (asphalt base).	Residual flux "C" (semi- paraffine).	Residual flux "D" (semi- paraffine).
Evaporations on 100 grams at 500° Fahrenheit:				
(a) Open, without stir- ring; loss.....				
Penetration of residue	16%	20%	33%	12%
Time.....	82	95	98	92
	1½ hrs.	10 hrs.	12¾ hrs.	10 hrs.
(b) Open, with stirring; loss.....				
Penetration of residue	16%	20%	33%	12%
Time.....	86	120	96	114
	1¾ hrs.	5 hrs.	5¾ hrs.	8½ hrs.
(c) In oven, without stirring; loss.....				
Penetration of residue	16%	20%	30.5%	12%
Time.....	73	49	97	300
	5 hrs.	19 hrs.	17 hrs.	3 hrs.

In the second table there are exhibited results of

evaporations on the same materials as in Table 1, but under various conditions. All the evaporations in Table 1 were made on 100 grams of asphalt in a container 5 inches in diameter by $2\frac{3}{8}$ inches deep. That there are very marked differences, regardless of the quantity and other data, and depending only on the difference in the various methods of heating is shown in Table 2, from which it can readily be seen that the oils of various bases behave very differently under varying conditions.

Therefore, it is necessary when specifying asphaltic contents, to always state the following:

1. Quantity of asphalt applied (the evaporation being a surface function, is of course largely dependent on the ratio of the surface to the volume, and will vary with varying quantities).

2. Size of vessel.

3. Temperature, and *length of time* to be exposed to the same; the latter item is evidently the most important one.

4. Whether the evaporation takes place.

(a) In a closed oven.

(b) In the open.

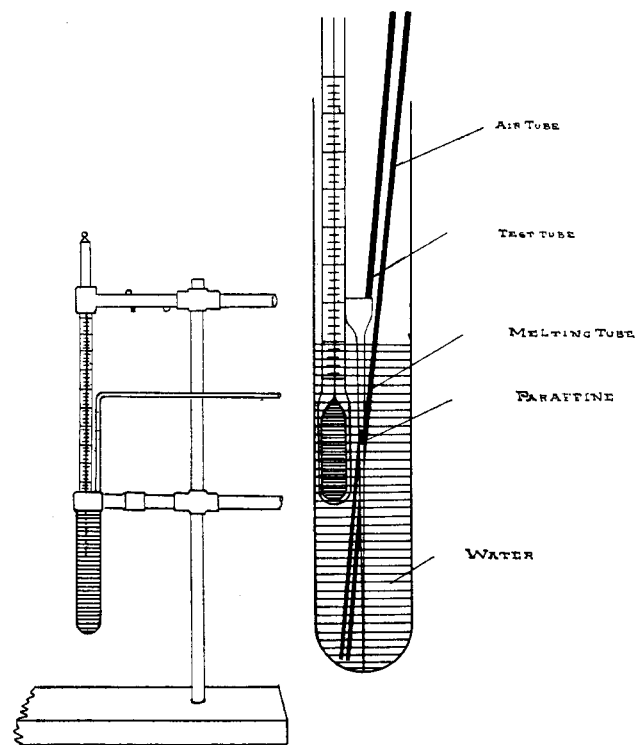
(c) With or without stirring.

2. *Paraffine*.—Next in importance to asphalt is paraffine as a distinguishing feature of petroleum. Without entering here into the question of merit or disadvantages of paraffine in asphalt, there has been a great deal of uncertainty regarding its determination. It should be remembered that paraffine is not a chemical individual, and that the percentage of crystallizing paraffine scale in a given distillate depends entirely on the solvent and also on the temperature at which this crystallizing takes place. It should also be remembered that paraffine is never contained in a crude oil or in a residual in its scaly modification, but always as amorphous, so-called "proto" paraffine. Therefore the first demand is to make the asphaltic material subject to destructive distillation to coke, and determine the scale in the distillate. In some asphalts there is no paraffine whatsoever and for this reason a qualitative test should be made first, as follows: The asphalt is distilled to coke, and a drop of the distillate is caught at intervals and put on ice. The temperature and percentage of distillate should be noted at the point when this drop chills.

Some asphalts furnish a distillate which chills at once, whereas others are practically free from paraffine scale; therefore a quantitative determination does not have to be made. The quantitative methods now in use are either the one by Zalociecki or that by Holde. Zalociecki dissolves the distillate in amyl alcohol and precipitates with ethyl alcohol at 0° to -5° F., whereas Holde precipitates with alcohol ether at -20° C.

Table 3 contains paraffine determinations of various asphalts and it will be seen at first sight that

the results, although taken on one and the same material, differ vastly. This is for the reason that, as above stated, paraffine is not paraffine in every case, and that the amount of paraffine scale crystallizing out is entirely dependent on its consistency. The colder the "wax distillate," the softer is the crystallizing scale; this is in accordance with the commercial process of bringing out paraffine wax, which is marketed and designated from different "pressings" by melting point. The writer has therefore made it a practice



Apparatus for determining melting point of paraffine.

to invariably determine the melting point of paraffine, and it cannot be demanded urgently enough that in all such present specifications which set a limit to paraffine scale, its melting point should also be given, as otherwise there would be no end of discrepancy. There seems to be a certain ratio between melting point and percentage, and in most cases a difference of 2° F., melting point, seems to be equivalent to one per cent. scale.

TABLE 3.—RELATION BETWEEN CONSISTENCY AND PERCENTAGE OF PARAFFINE.

Determinations:	1.	2.	3.	4.	
	Zal.	Zal.	Holde.	Holde.	
No. 1 (65% binder; residual)	16.5	15.5	4.5	Flows
Melting point.....	96° F.	104° F.	120° F.	
No. 2 (90% binder; residual).....	10.0	9.0	5.0	Flows
Melting point.....	108° F.	110° F.	118° F.	
No. 3 (95% binder; residual).....	6.2	4.3	2.7	Semi-fluid
Melting point.....	116° F.	122° F.	131° F.	
Compound No. 3.....	13.4	7.5	7.3	10.0	Liquid
Melting point.....	105° F.	124° F.	126° F.	118° F.	

For the melting point we use the capillary method. The results as exhibited in Table 2 speak for

themselves. It should be remembered in this connection that even the originators of the methods of determination do not claim accuracy within one per cent., whereas there are a number of asphalt specifications stipulating percentages within one-quarter of one per cent. and even less of paraffine; this is totally objectionable especially when no melting point is given.

There are also a number of specifications having a so-called "cold test," as follows:

"When a six inch test tube, having an inside diameter of one inch is filled one-half full of the oil, cooled to and maintained at 50° F., for a period of thirty minutes, then brought to and maintained at 65° F., for a period of thirty minutes, it must at that temperature (65° F.) show a flow perceptible to the eye in thirty seconds. The oil after cooling to 50° F. must not be stirred or agitated in any way before or during the flow test and after being cooled at 50° F. it must not be heated to a temperature exceeding 65° F., until after its flow has been determined."

Upon inquiring of the originators of this specification it was learned that this extremely complicated clause had the object of eliminating paraffine oils. It was stated that if such materials were to contain paraffine to any extent, they would chill at 65° F., thus not complying with the above specification. This sounds plausible enough but since we found that bitumens such as represented on Table 3, although all containing more than three per cent. paraffine, would show a flow according to this specification at 65° F., we have concluded to investigate this matter more thoroughly. A heavy Texas distillate, which contained neither paraffine nor asphalt, was taken as a neutral base. At first we dissolved in it 8 per cent. of paraffine scale of a melting point of 125° F. This compound represented a solid mass at room temperatures. We then added 8 per cent. of a soft Texas asphalt, and the result was compound No. 3, which is a thorough liquid, and more liquid even than the asphalt dissolved in the oil alone. We have, therefore, the case of a lowering of the melting point produced by the combined action of paraffine plus asphalt. Therefore, the only reason that binders 1, 2 and 3 (Table 3) are liquid at 65° F. as specified, can be sought for in the fact that they contain asphalt and paraffine. If one or the other were removed, the remainder would become harder. This we have also shown on a residual cylinder oil, of semi-asphaltic basis which contained some asphaltic matter; alcohol ether precipitated 18.5 per cent. The oil was thoroughly liquid (cold test = 75° F.). It was filtered through porous matter, which retained the asphaltic substance, and the result was a light-colored product which is solid at ordinary temperatures, its cold test being 90° F. Attached is an analysis of the oil before and after filtering.

This case illustrates conclusively that the consistency of an asphaltic compound alone can never give an idea of its true contents, and that a specification like the above is utterly worthless.

3. *Determination of Tar.*—It has so far been impossible to ascertain the presence of tar except by more than very doubtful methods. The method which is herewith introduced will give, if not extremely accurate ones, results of satisfactory commercial value. The principle of the method consists in distilling the bitumen to coke, and applying dimethylsulfate to the same. Dimethylsulfate dissolves oil products of benzene character or pyro distillates, whereas it does not dissolve paraffine or olefine hydrocarbons, such as produced by the distillation of mineral oil. E. Graefe has applied this reaction to separate brown coal or shale distillate from coal tar products, and the writer found that it represents an ideal means of separating all distillates obtained from pyro-bitumen, or all benzene derivatives from petroleum distillates, which, of course, consist largely of paraffine chains or other saturated hydrocarbons.

TABLE 4.—PERCENTAGE COAL TAR BY SOLUBILITY OF THE DISTILLATE IN DIMETHYLSULFATE.

Description of product.	Solubility in per cent
E Grade petrol. asphalt.....	Not soluble
Bermudez asphalt.....	" "
Cuban asphalt.....	" "
Gilsonite.....	" "
Coal tar.....	100 "
20% tar + 80% asphalt.....	15 "
40% " + 60% "	30 "
50% " + 50% "	35 "
60% " + 40% "	40 "
80% " + 20% "	60 "

Table 4 containing results obtained by this method gives the solubilities in dimethylsulfate of straight coal tar distillate, straight asphalt distillate (both native and oil), and various mixtures. The results illustrate that there is a certain fixed proportion by which we can very well judge the relative proportion of each. It is also interesting to note that all native asphalts, including gilsonite, act exactly like oil asphalts with the dimethylsulfate, which is a further proof of the identity of their origin. The importance of this method is considerable, inasmuch as the market value of tar is of course far below that of asphalt, and the lack of a method of determination has been felt very keenly. The method as executed in the writer's laboratory is as follows:

Four cc. of the distillate obtained by destructive distillation of the asphalt (to coke) are put in a ten cc. graduated cylinder with ground glass stopper, and six cc. of dimethylsulfate are added and shaken thoroughly for one minute. If the distillate is not entirely soluble, separation takes place within a few minutes and a separating mark can be accurately read. The percentage of solubility is calculated from this reading.

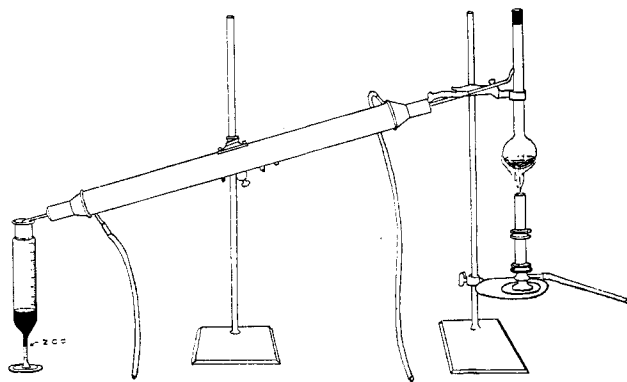
The methods outlined above may well be char-

acterized as an attempt to characterize and eventually analyze bitumina by their destructive distillate, which principle is new and I believe highly promising.

APPENDIX.

In the following we briefly describe a method of determination of water and one for the determination of sulphur. Both of these methods were originated by Edmund Graefe for crude oils and coal; the following describes their modification for asphaltic substances, for which they supply long-felt wants.

(a) *Water Determination.*—In distilling off water from heavy bodies such as bituminous matters or crude oil, foaming takes place which renders distillation entirely impossible. Graefe applies a light volatile oil, such as petroleum distillate or coal tar distillate, to the substance and distils both. In order to determine water in asphalt, 50 cc. of asphalt are mixed with 50 cc. of a petroleum distillate of volatile character. A product of this kind can be purchased in any quantities, it being a well-known turpentine substitute.¹ The total is then distilled and distillation proceeds easily and without any explosions. The same volume of the applied solvent is distilled off and thus made sure that all the water will be driven over, which, of course, settles after condensing. The combined distillates are caught in a flask having a

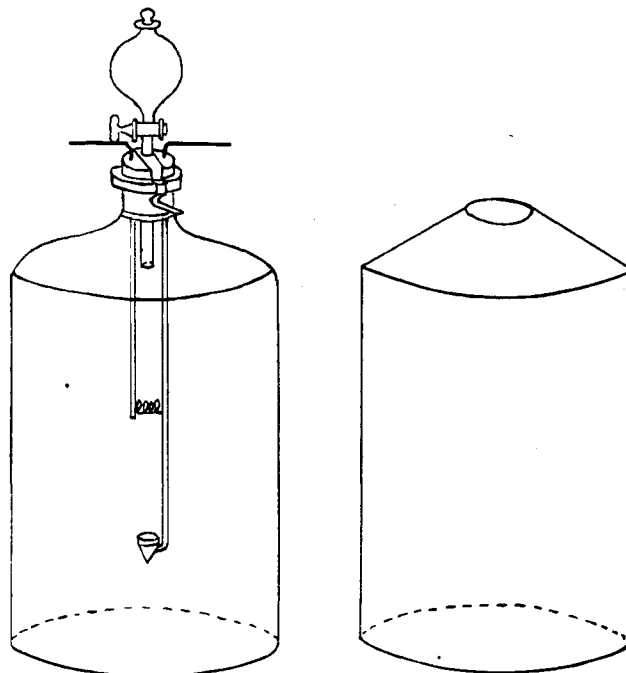


Apparatus for determination of water.

stem divided into 2 cc. This corresponds to one per cent. if 50 cc. of asphalt were applied, the water settling easily from the light distillate, and its percentage can be read at once, without any figuring.

(b) *Sulphur Determination.*—The principle of this method consists in the combustion of the bitumen in an atmosphere of oxygen. This was at first suggested by Hempel, and later modified by Graefe for coal products and was successfully applied in the author's laboratory on oils and asphalts. About 0.2 gram of bitumen are weighed and placed on a small lump of chemically pure cotton, which must be free from sulphur. This is then placed on a small platinum cone which, again, is suspended from a copper wire. The cotton containing the bitumen is connected to

a thin platinum wire, forming a short-circuit between the suspending copper wire and another wire through which an electric current can be sent coming from



Apparatus for determination of sulphur.

a dry battery. The current will thus ignite the thread, the cotton and finally the asphalt, which will burn freely in the oxygen. After combustion is completed a solution of sodium peroxide is permitted to enter the flask. Care should be taken that this be done either after the bottle is cold, or that the surplus pressure is released gradually through the solution of sodium peroxide in which case it would retain all sulphurous gases. Determination of sulphur is finally accomplished by neutralization and precipitation of the solution with barium chloride. With a sufficient number of apparatus it is easy to execute two dozen sulphur analyses per day. They are accurate almost to the theoretical point which has been established by a number of experiments.

THE DETERMINATION OF COPPER IN BLISTER AND REFINING COPPER.

By WILLIAM C. FERGUSON.¹

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Through the efforts of representatives of those interested in the commerce of copper, continual advances have been made in sampling and analysis with the object of securing its correct valuation.

Although the electrolytic method for the determination of copper is universally employed, marked differences exist in the detail of its performance.

A description of the method developed in the laboratory of the Nichols Copper Co. is, therefore, pre-

¹ The commercial name of this product is *Texene*. Its gravity is 42° Beaumé and its limits of evaporation are between 270° and 450° F.

¹ Chief Chemist, Nichols Copper Co.