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THE EVAPORATION TEST FOR MINERAL LUBRICATING AND TRANSFORMER OILS

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EVAPORATION TEST FOR MINERAL LUBRICATING AND TRANSFORMER OILS

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A year ago the difficulty in obtaining concordant duplicate determinations of the percentage of loss by evaporation when certain mineral oils were heated was forcibly impressed upon the writer. It was not until quite recently, however, that any attempt was made to determine to what extent the amount and rate of evaporation are influenced by conditions to be described below.

The results obtained with a typewriter oil and with two transformer oils finally led to the present work being done. The results lead to the conclusion that the factor of prime importance is, for a given temperature, the area of oil surface exposed to the atmosphere. Even when quite different weights of oil are heated in vessels of the same size, the actual losses are nearly equal, so that when the results are figured as percentages they are far from concordant. As far as we have been able to find from the literature, comparatively little attention has been paid to this factor and, indeed, to the whole subject of the evaporation of oil.

Gill [1] uses filter paper, 1⅝ inches in diameter with a five-eighths inch hole in the center. The paper is dried in a desiccator over sulphuric acid, weighed on a watch glass and then "about 0.2 gram" of oil is dropped upon it.

We have found that eight small drops of a certain oil was sufficient to saturate the paper. This amount of oil weighed from 0.1401 to 0.1506 g.

[1] Oil Analysis, 6th ed., p. 35.

Holde [2] uses the inner cup of the Pensky flash-point apparatus. The cup is filled to the mark and the amount of oil determined by weighing. With this cup, heated in the bath devised by Holde, it should be easy to duplicate results very exactly with a given oil. Since the loss in weight of oil depends so largely on the surface, it might seem that duplicate determinations of the percentage loss would not agree exactly if great care were not taken to fill the cup to the mark. As a matter of fact a variation of 1 mm in the depth of the oil makes a difference of a little more than 2.0 cc and, therefore, of rather less than 2.0 g. The standard transformer oil of the General Electrical Society of Berlin [3] has the specific gravity 0.8825 and does not lose more than 1.0 per cent on heating to 170° for two hours. Assuming the actual loss in weight to be the same in both cases, a difference of 1 mm in the depth of the oil causes a difference in the percentage loss of only 0.03 per cent.

Considerable differences in the specific gravities of two oils, assuming that they both lose the same amount when heated, cause only slight variations in the percentage losses. If the oil of specific gravity 0.8825 loses 1.0 per cent in weight, and one of specific gravity 0.8925 loses the same, the percentage loss in the latter case is 0.989 per cent. These figures are based on the dimensions of a new cup, 51.4 mm in inside diameter, with the mark 35 mm from the bottom.

The chief objections to the use of the Pensky cup are its weight and the considerable amount of oil needed.

Schreiber [4] first used the apparatus of Holde, but later [5] employed bacteriological culture dishes, "80 to 85 mm in diameter and 20 mm high." Here is introduced a variation of nearly 13 per cent in the area of oil exposed. In addition there must be variations in the thickness of the glass bottoms and walls.

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[2] Mitth. techn. Vers.-Anstalt, Berlin, **20**, pp. 67–70; 1902.

[3] Lunge: Chem.-Techn. Untersuchungsmeth., 5te. Aufl., Bd. III, p. 50.

[4] Zs. angew. Chem., **18**, pp. 726–734; 1905.

[5] Ibid., **23**, pp. 99–103; 1910.

Allen [6] is most indefinite in prescribing the conditions under which the evaporation test should be made. "A known weight should be placed in a watch glass, wide beaker or flat porcelain dish," and then heated. Again, "spindle oil should not lose more than 5 per cent of its weight when absorbed by filter paper" and then heated.

According to Archbutt and Deeley,[7] the volatility of an oil is commonly determined by heating 0.6 to 1.0 g in a shallow dish or watch glass. Archbutt, however, pointed out that the amount of evaporation must necessarily depend on the rate of change of the air in the oven. He devised an apparatus in which the oil is heated in a current of air or of steam, flowing at a constant rate and previously heated to the temperature of the bath. The oil (0.5 g) is placed in a platinum dish, 3 inches long, ½ inch wide, and ¼ inch deep. The heating bath is somewhat complicated.

Since the object of the work described in this paper was chiefly to determine the differences in the amounts of evaporation when a given weight of oil was heated with a greater or less surface area exposed to the atmosphere, a set of tubes was made from thin brass tubing with bottoms of thin sheet brass soldered on. The tubes were commercial sizes having inside diameters of 2, 3, 4 and 5 cm, respectively. Tubes both 3 and 5 cm in depth were made.

The tubes, containing weighed amounts of oil, were heated in an aluminium air bath covered with sheet asbestos. They rested on a plate of aluminium, about 3 mm thick, to distribute the heat more uniformly. There were four ventilating openings in the bottom of the bath and two in the top.

In Table I are given the results obtained on heating 5 g of an oil intended for use in automobile engine cylinders, in each of the shorter tubes and also in the pair of longer tubes 2 cm in diameter. This oil is designated as "A."

Throughout this paper the time of heating is counted from the moment the flame under the bath was lighted until the tubes were removed.

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[6] Coml. Org. Anal., ed. 2, 2, II, pp. 128 and 136.

[7] Lubrication and Lubricants, pp. 191–195.

TABLE I

Evaporation of Oil "A" at 110°

Weight taken, 5.00 g

Tube	A	B	C	D	E	F	G	H	I	J
Diameter and depth (cm)	2 x 3	2 x 3	2 x 5	2 x 5	3 x 3	3 x 3	4 x 3	4 x 3	5 x 3	5 x 3
Loss in 3 hours (mg)	8.5	8.8	8.1	8.2	8.7	9.0	9.7	10.3	11.5	11.4
Loss (per cent)	0.17	0.18	0.16	0.16	0.17	0.18	0.19	0.21	0.23	0.23
Loss in 5 hours more (mg)	1.7	1.6	0.8	0.8	3.8	3.7	5.7	6.0	10.4	9.0
Loss (per cent)	0.03	0.03	0.02	0.02	0.08	0.07	0.11	0.12	0.21	0.18
Loss in 6 hours more (mg)	1.2	1.4	0.3	0.4	2.9	3.8	6.2	5.8	Lost	9.4
Loss (per cent)	0.02	0.03	0.01	0.01	0.06	10.08	0.12	0.12	.....	0.19
Total loss (mg)	11.4	11.8	9.2	9.4	15.4	16.5	21.6	22.1	.....	29.8
Total loss (per cent)	0.23	0.24	0.18	0.19	0.31	0.33	0.43	0.44	.....	0.60

It was thought advisable to repeat this series of determinations under exactly the same conditions. The results are given in Table II.

TABLE II

Evaporation of Oil "A" at 110°

Weight taken, 5.00 g

Tube	A	B	C	D	E	F	G	H	I	J
Diameter and depth (cm)	2 x 3	2 x 3	2 x 5	2 x 5	3 x 3	3 x 3	4 x 3	4 x 3	5 x 3	5 x 3
Loss in 3 hours (mg)	9.0	8.9	7.9	8.8	9.4	9.4	10.3	10.1	11.8	11.4
Loss (per cent)	0.18	0.18	0.16	0.18	0.19	0.19	0.21	0.20	0.24	0.23
Loss in 5 hours more (mg)	1.7	1.3	0.6	0.3	2.1	3.3	6.1	4.9	10.7	10.4
Loss (per cent)	0.03	0.03	0.01	0.01	0.04	0.07	0.12	0.10	0.21	0.21
Loss after 6 hours more (mg)	Lost	1.4	0.8	1.1	3.3	3.2	5.3	6.1	8.8	11.6
Loss (per cent)	.....	0.03	0.02	0.02	0.07	0.06	0.11	0.12	0.18	0.23
Total loss (mg)	.....	11.6	9.3	10.2	14.8	15.9	21.7	21.1	31.3	33.4
Total loss (per cent)	.....	0.23	0.19	0.20	0.30	0.32	0.43	0.42	0.63	0.67
Averages, Tables I and II	0.23		0.19		0.32		0.43		0.63	

From the results given in these two tables it is evident that the amount of evaporation, though small in any case, is greater the larger the tube used. The figures showing the losses during the first 3 hours probably largely represent loss of moisture and a little

volatile oil. During the next two periods of heating the differences between tubes of successively greater diameter stand out more clearly. The influence of the depth of the tube is also shown by the results obtained with tubes A and B (3 cm deep) as compared with tubes C and D (5 cm deep), all of which are of the same diameter.

A sample of transformer oil (oil "B") was found to show much greater losses on heating, besides bringing out more clearly the increase in the amount evaporated as the area of the oil surface increased. The results are given in the following table:

TABLE III

Evaporation of Oil "B" at 110°

Weight taken, 5.00 g; Depth of tubes, 3 cm

Tube	A	B	E	F	G	H	I	J
Diameter of tube (cm)	2	2	3	3	4	4	5	5
Loss in 3 hours (mg)	15.5	19.5	25.7	29.1	66.1	64.5	126.4	112.6
Loss (per cent)	0.31	0.39	0.51	0.58	1.32	1.29	2.53	2.25
Loss in 3 hours more (mg)	18.6	15.4	26.0	37.8	63.9	70.0	128.2	117.3
Loss (per cent)	0.37	0.31	0.52	0.76	1.28	1.40	2.56	2.35
Loss in 3 hours more (mg)	18.2	16.3	48.4	42.4	88.6	84.0	119.7	130.0
Loss (per cent)	0.36	0.33	0.97	0.85	1.77	1.68	2.39	2.60
Loss in 3 hours more (mg)	18.7	20.6	38.7	42.1	75.2	74.8	113.8	127.4
Loss (per cent)	0.37	0.41	0.77	0.84	1.50	1.50	2.28	2.55
Total loss (mg)	71.0	71.8	138.8	151.4	293.8	293.3	488.1	487.3
Total loss (per cent)	1.40	1.44	2.78	3.03	5.88	5.87	9.76	9.75

Tubes C and D (5 cm deep) were heated at the same time as the shorter ones, and gave such low results that tubes 5 cm deep of each of the larger sizes were made. In Table IV are given the results of two successive runs, using these deeper tubes, and including the results obtained when C and D were heated with the shorter tubes.

TABLE IV

Evaporation of Oil "B" at 110°

Weight taken, 5.00 g; Depth of tubes, 5 cm

Tube	C	C	C	D	D	D	E′	E′	G′	G′	I′	I′
Diameter (cm)	2	2	2	2	2	2	3	3	4	4	5	5
Loss in 3 hours (mg)	3.0	7.1	9.0	2.6	6.6	8.6	15.0	19.7	62.9	74.4	124.8	152.9
Loss (per cent)	0.06	0.14	0.18	0.05	0.13	0.17	0.30	0.39	1.26	1.49	2.50	3.06
Loss in 3 hours more (mg)	2.4	5.6	6.2	1.9	5.3	6.6	12.4	18.7	56.8	71.6	126.8	137.3
Loss (per cent)	0.05	0.11	0.12	0.04	0.11	0.13	0.25	0.37	1.14	1.43	2.54	2.75
Loss in 3 hours more (mg)	3.3	6.0	5.2	3.2	5.9	5.7	16.8	19.1	67.9	67.0	129.4	124.9
Loss (per cent)	0.07	0.12	0.10	0.06	0.12	0.11	0.34	0.38	1.36	1.34	2.59	2.50
Loss in 3 hours more (mg)	5.3	6.9	4.6	4.5	6.9	4.6	20.6	14.6	81.8	38.9	113.5	87.5
Loss (per cent)	0.11	0.14	0.09	0.09	0.14	0.09	0.41	0.29	1.64	0.78	2.27	1.75
Total loss (mg)	14.0	25.6	25.0	12.2	24.7	25.5	64.8	72.1	269.4	251.9	494.5	502.6
Total loss (per cent)	0.28	0.51	0.50	0.24	0.49	0.51	1.30	1.44	5.39	5.04	9.89	10.05

The results, while not as concordant as one could wish, show clearly, when compared with those in Table III, that with increasing depth of tube there is a lower rate of evaporation, except for the tubes of the greatest diameter. If the lower rate of evaporation in the deeper tubes is due to condensation on the upper part of the walls or to a hindering of the convection currents carrying away oil vapors, we should expect the effect to be least in the larger tubes, because a smaller proportion of the vapors comes in contact with the walls and there is more space for the free circulation of convection currents flowing in opposite directions. By the end of the third or fourth heating there were small amounts of oil around the outside of the mouths of the tubes, but no difference in this respect was noticed between the tubes of less or greater diameter, so that it would seem to be due to the ordinary "crawling" of the oil. From this, and from the excellent heat conductivity of brass, it seems that the hindering of the convection currents plays the principal part. The only practical way to avoid this effect is to have the oil vessels sufficiently broad and shallow.

As mentioned above, there was some evidence obtained months ago, tending to show that when quite different weights of the same oil are heated in vessels of the same size the losses are about the

same when expressed in milligrams. To test this further, a series of determinations was made in the brass tubes 3 cm in depth, using 1.0 g of oil instead of 5.0 g, as in the experiments described above. The two deeper tubes, C and D, were also used. The results are in the following table:

TABLE V

Evaporation of Oil "B" at 110°

Weight taken, 1.00 g

Tube	A	B	C	D	E	F	G	H	I	J
Diameter and depth (cm)	2 x 3	2 x 3	2 x 5	2 x 5	3 x 3	3 x 3	4 x 3	4 x 3	5 x 3	5 x 3
Loss in 3 hours (mg)	7.0	8.7	3.1	3.4	26.6	35.1	79.0	80.5	121.4	156.4
Loss (per cent)	0.70	0.87	0.31	0.34	2.66	3.51	7.90	8.05	12.14	15.64
Loss in 3 hours more (mg)	8.5	7.6	3.0	2.8	39.6	31.9	67.8	68.4	105.2	85.4
Loss (per cent)	0.85	0.76	0.30	0.28	3.96	3.19	6.78	6.84	10.52	8.54
Loss in 3 hours more (mg)	7.0	7.4	3.4	3.1	31.2	34.5	58.8	55.5	70.7	74.2
Loss (per cent)	0.70	0.74	0.34	0.31	3.12	3.45	5.88	5.55	7.07	7.42
Loss in 3 hours more (mg)	5.4	7.8	3.1	3.0	22.9	28.8	54.1	40.6	55.6	47.0
Loss (per cent)	0.54	0.78	0.31	0.30	2.29	2.88	5.41	4.06	5.56	4.70
Total loss (mg)	27.9	31.5	12.6	12.3	120.3	130.3	259.7	245.0	352.9	363.0
Total loss (per cent)	2.79	3.15	1.26	1.23	12.03	13.03	25.97	24.50	35.29	36.30
Averages (per cent)	2.97		1.25		12.53		25.24		35.79	
Averages on basis of 5 g oil (per cent)	0.59		0.25		2.51		5.05		7.16	

The percentage losses are very much larger than when there was 5.0 g of oil in each tube. The actual losses in milligrams, or what amounts to the same thing, the percentage losses calculated on the basis of 5.0 g of oil, may be compared with the figures in Table III and with tubes C and D in Table IV.

As might have been predicted, the losses in the tubes 2 cm in diameter (A to D) are less with one than with 5 g of oil, on account of the relatively much longer air space in the tubes above the oil. A and B (Table V) when containing 1.0 g have, however, about the same air space as C and D (Table IV) when holding 5.0 g, and we find the losses in milligrams, roughly, the same.

With the tubes 3 cm in diameter, E, F, and E′, the losses are fairly comparable, showing the lessening influence of the air space

in the tubes. With the tubes 4 cm in diameter, G, H, and G′, and still more clearly with the 5-cm tubes, I, J, and I′ (Tables III and V), we can see that the single gram of oil taken is approaching its limit of volatility at 110°, the total loss being naturally less than when 5.0 g of oil was used. The losses in milligrams for the first six hours' heating for each size of tube above 2 cm diameter are about the same, respectively, whether 1.0 or 5.0 g of oil was taken (Tables III and V).

A series of determinations was made using Gill's method, described above. The eight drops of oil used each time did not weigh "about 0.2 g," but the amount was sufficient to thoroughly saturate the ring of filter paper. The more volatile oil "B" was first heated. The results follow:

TABLE VI

Evaporation of Oil "B" at 110°

Gill's Method

Weight of oil (g)	0.1441	0.1490	0.1401	0.1436	0.1506	0.1409
Loss in 3 hours (mg)	45.6	55.3	44.8	49.4	46.2	43.1
Loss (per cent)	31.64	37.11	31.98	34.40	30.68	30.59

Four determinations were also made on oil "A," together with two more on oil "B," for comparison.

TABLE VII

Evaporation of Oils "A" and "B" at 110°

Gill's Method

Oil	A	A	A	A	B	B
Weight of oil (g)	0.1431	0.1327	0.1407	0.1416	0.1407	0.1464
Loss in 3 hours (mg)	2.9	0.9	2.1	2.7	45.0	49.1
Loss (per cent)	2.03	0.68	1.49	1.91	31.98	33.54
Loss in 3 hours more (mg)	1.5	3.1	1.8	1.9	19.1	20.7
Loss (per cent)	1.05	2.34	1.28	1.34	13.58	14.14
Total loss (mg)	4.4	4.0	3.9	4.6	64.1	69.8
Total loss (per cent)	3.08	3.01	2.77	3.25	45.56	47.68

A final series of determinations was made with oil "B," using filter papers 1⅝ inches in diameter, (*a*) without holes cut in them, (*b*) with ⅝-inch holes, and (*c*) with $\frac{29}{32}$-inch holes, respectively. Since six drops of oil saturated the last papers, this amount was used throughout.

TABLE VIII

Evaporation of Oil "B" at 110°

Gill's Method with Modifications

Papers	No holes			⅝-inch holes			$\frac{29}{32}$-inch holes		
Weight taken (g)	0.1124	0.1104	0.1063	0.1142	0.1061	0.1023	0.1034	0.1021	0.1063
Loss in 3 hours (mg)	36.1	36.3	36.9	44.5	47.6	40.9	37.1	41.2	34.7
Loss (per cent)	32.12	32.88	34.71	38.97	44.86	39.98	35.88	40.35	32.64
Loss in 3 hours more (mg)	13.1	10.4	11.6	9.7	7.8	8.5	11.4	10.6	9.9
Loss (per cent)	11.65	9.42	10.91	8.49	7.35	8.31	11.03	10.38	9.31
Loss in 3 hours more (mg)	12.2	14.2	10.0	9.8	10.9	10.4	9.2	9.8	9.7
Loss (per cent)	10.85	12.86	9.41	8.58	10.27	10.17	8.90	9.60	9.13
Total loss (mg)	61.4	60.9	58.5	64.0	66.3	59.8	57.7	61.6	54.3
Total loss (per cent)	54.62	55.16	55.03	56.04	62.49	58.46	55.81	60.33	51.08

With an oil of comparatively high volatility it appears that the results obtained by heating on filter paper are not to be compared, as far as their concordance is concerned, with those obtained by heating in brass tubes. The total losses, in milligrams, observed with a less volatile oil ("A" in Table VII) are much more concordant, but when calculated as percentages they may show wide variations unless care is taken to weigh out always exactly the same quantity.

Although in the above experiments the oil was always heated to 110°, it is believed that the differences would be of the same order at lower or higher temperatures. The only object in heating the oil is to hasten an operation that must be taking place at ordinary temperatures, though much more slowly. In this connection may be given the results of some experiments made on a typewriter oil many months ago. It was this, indeed, which first showed clearly the need of very exact duplication of all of the conditions.

Portions of the oil were heated in 50– cc Jena beakers and in ordinary weighing bottles of smaller diameter than the beakers. The dimensions were not recorded.

TABLE IX

Evaporation of Typewriter Oil

Vessel	Beaker		Weighing bottle	
Weight of oil (g)	0. 6027	0. 8666	0. 6786	0. 8610
Loss in 5 hours at 55° (mg)	6. 5	7. 3	4. 0	5. 3
Loss (per cent)	10. 78	8. 42	5. 90	6. 16
Loss in 4 hours more at 65° (mg)	9. 4	9. 8	3. 3	3. 7
Loss (per cent)	15. 60	11. 31	4. 86	4. 30
Total loss (mg)	15. 9	17. 1	7. 3	9. 0
Total loss (per cent)	26. 38	19. 73	10. 76	10. 45

These figures pointed so clearly toward the need of having equal surface areas of oil exposed that two determinations were made, using 5.5– cm filter papers in wide weighing bottles.

TABLE X

Evaporation of Typwriter Oil at 55°

Weight of oil (g)	0. 5835	0. 6837
Loss in 3½ hours (mg)	13. 5	13. 6
Loss (per cent)	2. 31	1. 99
Loss in 3 hours more (mg)	6. 1	6. 1
Loss (per cent)	1. 05	0. 89
Total loss (mg)	19. 6	19. 7
Total loss (per cent)	3. 36	2. 88

Finally, samples of the oil were exposed at room temperature in wide weighing bottles covered with filter paper to exclude dust.

TABLE XI

Evaporation of Typewriter Oil at Room Temperature

Weight of oil, Feb. 21 (g)	0. 6066	0. 7439
Loss by Mar. 1 (mg)	0. 8	0. 8
Loss (per cent)	0. 13	0. 11
Further loss by Apr. 3 (mg)	6. 1	6. 0
Loss (per cent)	1. 01	0. 80
Total loss (mg)	6. 9	6. 8
Total loss (per cent)	1. 14	0. 91

It would seem as if sufficient evidence has been presented in this paper to show that, in order to obtain comparative results, the same weight of oil must always be heated in vessels of the same size, so that the oil surface shall always be the same in area and the convection effects be alike. Brass vessels are preferable to those of glass, because they can be made with their walls and bottoms of the same thickness, thus insuring more uniform heating. A convenient size is 5 cm in internal diameter with sides 3 cm high. Tubing of this size with a wall thickness of 0.75 mm can be bought. The bottoms may be made of sheet brass not more than 0.5 mm thick. It is best to use silver solder, so that the heating need not be limited to the lower temperatures. A vessel of the size indicated weighs somewhat less than 42 g.

A convenient weight of oil is 5.0 g. To avoid smearing oil on the walls of the tubes the writer used a small pipette with a 2-cm stem below the bulb. The final adjustment of the weight was made by just touching an oily or a dry stirring rod to the surface of the oil. It is comparatively easy to weigh out the oil within 0.5 mg of the amount desired, though a much larger variation would be of little consequence in calculating the percentage of evaporation.

In a neutral atmosphere there might have been somewhat greater losses, and possibly still greater differences between the losses with increasing diameter of tube, than the amounts given in the tables above. In the air there is more or less oxidation, partly involving loss of carbon dioxide and water, but mainly due to the formation of compounds containing carbon, hydrogen and oxygen, which are precipitated in part from the oil as a fine, brown sediment. The observed losses are really the sum of volatile oil, carbon dioxide and water lost, minus the oxygen taken up.

It would be interesting to repeat the work herein described, using an atmosphere of carbon dioxide, nitrogen or steam instead of air, but the results would be of less general application.

WASHINGTON, January 1, 1913.