

5. Error due to products of fermentation in old or spoiled samples is avoided by boiling before precipitating.

6. With the average moisture content of 10-12 per cent. for sugars, and 33-35 per cent. for sirups, the lead values are in each case on about the same amount of dry substance. It might be a good plan to make a moisture determination on each sample and calculate the *lead values* to "dry substance."

BUFFALO LABORATORY.

TYPEWRITER CARBON PAPERS.

By A. M. DOYLE.

Received June 20, 1908.

Typewriter carbon papers are much used in the departments of the government and for this reason an investigation into their qualities was made by the Contracts Laboratory of the Bureau of Chemistry. The paper foundation was tested for tensile strength, length and width; stretch under strain, both length and width; weight, inked and clean; thickness of inked paper; kind of pulp used and degree of beat. The ink, as distinct from the paper, was separated into its constituents of oil or wax, dye and pigments, and moisture and ash were determined. The written matter was rated on a scale of ten and was also subjected to sunlight and solubility tests.

Of the twenty-eight samples examined, five were copy papers and the others record. Copy papers included black, blue, purple and green, all of standard weight; record papers consisted of black, blue and purple; full-inked and semi-inked; standard weight, light weight and feather weight. Tables I, II and III represent the three classes of results. In Table I the physical tests have been arranged according to the weight of the paper, as described by the manufacturer, while in Tables II and III they are divided first into record and copy; then according to color—black, blue and purple, in the order named; and finally, within each group according to weight, from standard to feather weight.

Discussion of Results.

A comparison of the figures for the weight of the paper per square centimeter indicates that No. 2292 should be classed with light weight papers, Nos. 2290, 2293, 2294, 2301 and 2302 with standard weight, and Nos. 2300, 2381 and 2382 with the light weight; but otherwise these papers, grouped as indicated by the manufacturers on the label of the package, follow closely certain weight limits. Inked papers average less than 2 mg. per square centimeter for feather weight, from 2.2-2.4 mg. for light weight and a little less than 3 mg. for standard weight. The cleaned papers, free of ink, weigh less than 1 mg. for feather weight,

TABLE I.—PHYSICAL TESTS.

No. of sample.	Kind.	Wt. sq. cm. inked, mgms.	Wt. sq. cm. clean, mgms.	Tensile strength. Length, tenths, kgms.	Tensile strength. Width, tenths, kgms.	Stretch length, cm.	Stretch width, cm.	Thickness with ink, mm.	Kind of pulp.	Degree of beat.
2299	Standard weight	3.22	1.62	15.1	1.11	0.032	Rag, trace of ²	Fine
2303	"	3.05	1.70	15.0	0.81	0.039	Sulphite, trace of rag	Medium
2377	"	2.72	1.66	21.1	1.91	0.034	Rag, much ²	Coarse
2379	"	2.64	1.72	21.2	2.03	0.034	" some ²	"
2305	"	2.83	1.65	18.8	1.55	0.033	" some ²	Medium
2378	"	3.19	1.65	21.5	2.02	0.039	" some ²	Coarse
2380	"	3.02	1.52	16.8	1.37	0.040	" some ²	"
2304	"	3.85	1.64	16.2	1.02	0.045	Sulphate, trace of rag	Medium
2306	"	3.01	1.60	17.1	5.9	1.39	1.43	0.038	Rag, much ²	Coarse
2291c	"	2.90	1.65	17.3	1.31	0.034	"	Medium
2292	"	3.06	1.08	14.6	1.54	0.037	"	Fine
2295	"	2.92	1.57	13.3	6.2	0.65	0.81	0.042	Sulphite, little rag	"
2291d	"	3.10	1.61	17.9	1.37	0.033	Rag, little ²	Medium
Averages		3.04	1.64	17.5		
2289	Light weight	2.39	1.10	14.5	6.5	1.32	1.44	0.028	Rag	Fine
2290	"	2.40 ¹	1.71 ¹	18.1 ¹	11.0	1.79	1.70	0.031	" little ²	"
2291a	"	2.20	1.04	12.5	5.2	1.01	0.93	0.025	"	"
2291b	"	2.15	1.02	12.8	5.6	1.10	1.10	0.025	"	"
2293	"	2.61 ¹	1.67 ¹	16.8 ¹	9.3	1.39	1.33	0.026	" some ² and sulphate	Medium
2294	"	2.60 ¹	1.72 ¹	22.1 ¹	10.2	1.82	2.00	0.033	" some ²	"
Averages		2.39	1.06	13.6		
2288	Feather weight	1.90	1.04	14.6	1.23	0.018	Rag	Fine
2296	"	1.89	0.94	12.9	5.2	1.42	0.61	0.023	"	"
2297	"	1.81	0.92	13.9	1.60	0.021	"	"
2298	"	1.82	0.92	13.5	1.66	0.023	"	"
2300	"	2.62	1.15	15.7	1.80	0.028	"	"
2381	"	2.68	1.09	12.9	1.13	0.037	" some sulphite	Medium
2302	"	3.34 ¹	1.49 ¹	12.7	1.27	0.025	"	Fine
2382	"	2.93	1.07	14.0	1.32	0.037	" little sulphate	Medium
2301	"	3.37 ¹	1.64 ¹	15.6	1.76	0.028	"	Fine
Averages		2.23	1.02	13.9		

¹ Figures omitted in averages² Fiber resembling esparto.

between 1.0 and 1.1 mg. for light weight and from 1.6-1.7 for standard weight papers.

Mr. B. J. Howard, Chief of the Microscopic Laboratory, made all of the tests for thickness, kind of pulp and degree of beat. From the last column of the table it may be seen that the degree of beat of the pulp is generally coarse or medium in standard weight material and for the light and feather weight is usually fine or medium. The kind of pulp is not uniform, being either all rag, all sulphite, mixed rag and sulphite or mixed rag with a kind of fiber "somewhat resembling esparto, though without its characteristic structure." These fibers Mr. Howard found it "impossible to determine, as they were so thoroughly beaten as to almost destroy their identity. It was found impossible to distinguish between linen and cotton fibers for the same reason, and both together are classified as rag." In general, those papers containing rag fibers together with the undetermined fiber like esparto gave the highest tests for tensile strength and stretch. The thickness of the papers measured in thousandths of a millimeter showed decided average differences for the three weights, ranging from twenty for feather weight to twenty-five or thirty for light weight and from thirty-five to forty for standard weight.

Marked differences between papers of the same class were brought out by the tensile strength and stretch tests. A Schopper paper-testing machine adapted for weights from one to fifty kilograms was found to be too heavy, so a pendulum exercising one-fifth the strain was substituted on the same machine and all of the tensile strength tests were made with this smaller weight. The figures, as reported in the table, are, however, recalculated so as to be comparable with those obtained on the standard Schopper machine. In a few instances the tests were applied across fiber, as shown in columns on the table. The strip of material employed for the tests was 18 cm. in length and 1.5 cm. in width—the regulation size for this machine.

From the number of samples compared and the fact that their age was not known, it is difficult to trace any general rule with regard to the relation of thickness, kind of pulp and degree of beat with the tensile strength and stretch. Neither does the weight of the paper have the direct influence that might be expected. It is certain, however, that standard weight papers of approximately 1.65 mg. weight per sq. cm. should not be of a strength less than 18 tenth-kilograms (the unit of weight used on the table); nor have a stretch of less than 2 cm. in an 18 cm. strip. Similarly, for light and feather weight papers averaging 1 mg. weight per sq. cm., the strength and stretch should not fall below 14 tenth-kilograms and 1.5 cm. respectively. These tests bear directly on the wearing qualities of the paper and are valuable in making an estimate of its length of service.

TABLE II.—CHEMICAL TESTS.

RECORD PAPERS: BLACK.									
No.	Moisture. Per cent.	Ash. Per cent.	Carbon. Per cent.	Dye. Per cent.	Pigment. Per cent.	Wax. Per cent.	Ink. Per cent.	Paper. Per cent.	
2288	4.03	3.92	3.31	0.46	5.30	31.34	44.58	55.42	
2289	4.08	8.60	6.65	2.45	12.79	29.10	55.08	44.92	
2290	5.25	4.50	2.59	0.50	6.07	15.18	29.57	70.43	
2377	4.42	5.69	6.81	0.72	8.81	18.86	39.19	60.81	
2379	4.41	5.88	6.28	0.83	8.58	15.77	35.14	64.86	
2381	2.98	9.83	9.89	0.77	14.38	31.27	59.47	40.53	
2291a	3.73	5.20	5.53	1.20	8.30	31.87	50.92	49.08	
2291b	3.94	5.20	5.63	1.90	7.61	31.44	50.57	49.43	
2299	3.46	7.07	9.00	0.82	8.87	27.42	49.64	50.36	
2300	3.86	7.31	8.58	10.24	32.85	55.95	44.05	
2303	4.16	6.56	5.76	0.60	12.85	20.95	44.40	55.60	
2293	4.84	0.81	5.10	0.78	1.40	27.60	39.70	60.30	
2296	3.17	4.60	3.58	5.93	36.88	50.32	49.68	
2297	3.40	4.34	3.58	5.47	36.54	49.32	50.68	
2298	3.47	4.41	3.50	5.38	34.80	47.52	52.48	
BLUE.									
2294	5.58	9.11	1.51	13.13	18.78	39.00	61.00	
2302	3.16	14.36	1.47	25.03	25.05	55.38	44.62	
2305	3.78	11.28	0.53	18.76	19.04	42.25	57.75	
2378	4.19	13.76	0.83	25.52	18.09	48.11	51.89	
2380	3.79	17.39	0.77	26.15	18.80	49.70	50.20	
2382	3.57	21.87	0.87	26.77	30.34	62.98	37.02	
PURPLE.									
2301	3.65	14.69	22.66	5.28	19.71	51.28	48.72	
2304	3.87	16.56	25.50	7.69	19.66	57.22	42.78	
COPY PAPERS.									
2306	5.40	2.73	2.53	6.13	3.82	27.87	45.82	54.18	
2291c	4.34	1.53	4.42	3.74	30.15	43.10	56.90	
2292	3.45	2.26	6.52	6.25	37.07	63.80	36.20	
2295	4.83	2.64	11.51	4.60	25.11	46.05	53.95	
2291d	4.16	4.50	6.32	2.48	34.96	48.11	51.89	

The kind and relative amounts of the components of the ink are important factors in the working qualities of a typewriter carbon paper. The data obtained in Table II are useful in arriving at a general idea of the various ingredients used for ink, their relative proportions and those combinations which appear to give the best results in working qualities. The influence of the kind and state of division of the carbon and other pigments used, of the kind of oil or wax, of varying kinds and amounts of dyes, etc., can be determined only by an examination of a very large number of papers, the variation being so great that it is not now possible to present a formula for the composition of a standard ink. To illustrate the variation, the amount of ink in one of the best papers amounts to 63 per cent. and in another paper equally as good, so far as working qualities, there is but 43 per cent. of ink, while the average is between

50 and 55 per cent. Of course, in deciding between two papers that differ so widely as these mentioned, the important question is as to which has the larger amount of ink available for writing purposes.

For the base of the ink, paraffin waxes of rather high melting points are very generally employed, although not exclusively so. Pigments like Prussian blue and lampblack and dyes such as methylene blue and methyl violet appear in many of the papers, while in others the same or better results are obtained by other combinations. The percentage figures shown in Table II are of value mainly in correlation with the results shown in Tables I and III.

Methods.—Separation of the ink from the paper was difficult to accomplish. Several methods were tried. Boiling water, acids and alkalis poured over the paper after it had been cut into strips, separated the ink effectually, but disintegrated the paper. Ethyl ether or petroleum ether of low boiling point would not dissolve the wax of the ink so that it might be filtered. Petroleum ether of higher and definite boiling point was very hard to obtain, as ten gallons of 66° gasoline yielded but three quarts of a product boiling between 70° and 90°, and this was very hard to evaporate off completely as well as being dangerous to store and handle. Pure benzene, boiling point 81–2°, was finally adopted as the solvent, but it had to be prepared free from alcohol and water, so that dyes would not be affected. The benzene was therefore treated in the way ordinarily used for cleansing ether, by shaking repeatedly with water and then allowing it to stand for several days over metallic sodium.

A weighed portion of the paper, cut into narrow strips and put in a beaker, was covered with the prepared benzene, heated quickly on the steam-bath with shaking and poured through a weighed porcelain Gooch crucible, using suction. Three or more applications of the solvent removed all of the ink, possibly with the exception in some cases of a trace of dye. The filtrate, which was clear and usually colorless or pale yellow, was received in a weighed beaker, the solvent evaporated off, and the weight of the wax determined directly. The residue was afterwards examined to determine its properties, such as melting point, specific gravity, saponification value and iodine number.

Both the Gooch crucible, containing the pigment and dye, and the residue of paper were dried and weighed. They were then treated with hot alcohol of 95 and 70 per cent. strengths, and the Gooch in addition was thoroughly washed with hot water. Gooch and paper were again dried and weighed, the total loss being the weight of the dye. The solution of the dye in alcohol and water, thus obtained, was examined to determine the kind of dye employed. The weight of the paper, which was now perfectly clean, was recorded and the weight of total ink found by difference. The results, expressed as per cents., are found in Table II.

The Gooch crucible was then treated several times with hot alkalis and acids and the dried residue, if any, weighed as carbon. If, however, on burning off the carbon, an appreciable amount of ash still remained, the presence of insoluble pigments was shown, the kind and quality of these being determined by an examination of the ash of the whole paper.

Moisture and ash were determined on another portion of the sample; moisture at 100° to constant weight and ash at a low red heat. The ash was examined qualitatively and quantitatively for metals such as iron, chromium, aluminum, zinc, calcium, magnesium, etc., and for phosphoric, sulphuric, silicic acids, etc.

TABLE III.—TESTS OF WRITING.

No.	Original ranking.	After 5 days sun.	After 10 days sun.	Water.	1/2% HCl in water.	1/2% HCl in alcohol.	2% NH ₃ in water.	2% NH ₃ in alcohol.	^{n/100} Ca(OCl) ₂ in water.
RECORD PAPERS: BLACK.									
2288	1	2	3	2	2	2	4	2	3
2289	2	3	4	3	3	3	6	3	3
2290	5	6	6	6	6	6	8	6	6
2377	2	3	5	4	4	4	5	3	3
2379	2	3	5	4	5	5	5	3	3
2381	2	3	5	6	4	4	6	3	3
2291a	1	2	3	2	3	3	6	3	3
2291b	1	2	3	2	3	3	6	3	3
2299	4	5	6	5	6	5	7	5	5
2300	1	3	4	3	4	3	7	3	3
2303	2	2	3	5	3	3	5	3	3
2293	2	3	5	3	3	4	5	4	3
2296	2	3	5	3	3	4	5	3	3
2297	2	4	6	3	4	4	5	3	4
2298	4	3	5	5	4	5	4	5	5
BLUE.									
2294	4	5	7	5	5	6	8	8	5
2302	2	3	3	3	3	3	4	3	2
2305	3	4	6	4	4	4	5	5	4
2378	2	4	5	3	4	4	6	4	4
2380	2	4	6	5	4	4	8	4	3
2382	2	3	4	4	4	3	6	4	3
PURPLE.									
2301	2	7	8	3	5	6	7	6	3
2304	3	6	8	4	5	7	6	5	4
COPY PAPERS.									
2306	3-5-8	4-5-8	5-6-9	5-5-9	4-5-10	4-5-10	6-6-10	3-4-9	6-6-7
2291c	2-2-6	2-3-7	4-4-8	6-6-7	3-4-5	7-7-8	7-8-8	8-8-9	5-6-7
2292	6-6-8	6-7-9	7-8-10	7-8-8	5-6-7	8-9-9	7-8-8	7-8-8	6-7-8
2295	3-5-6	3-5-7	6-5-8	5-6-9	6-7-9	8-7-10	7-7-9	7-4-8	5-5-7
2291d	3-5-5	2-4-6	3-4-7	6-7-9	5-5-9	8-7-9	8-8-9	8-5-9	6-6-7

The writing used for these tests was made with a fresh sheet of the

copy paper. It was first rated on a scale of ten according to the following outline:

1. Clear, strong.
2. Almost as good as 1.
3. Not very clear nor strong, or strong but some blurred.
4. Between 3 and 5.
5. Lighter than 4 but still clear, or strong but much blurred.
6. Between 5 and 7.
7. Very light but clear, or strong and very badly blurred.
8. Between 7 and 9.
9. Very pale and weak, or much blurred, letters not distinct.
10. Very pale, almost illegible, or very much blurred, almost illegible.

It may readily be seen that characters can grade off from those that are clear, strong and perfectly satisfactory in either of two directions. They may become fainter, and thus fail in strength and clearness, or they may become heavier and blurred. The figures in Table III show that this is a ready means of separating the good papers from the poorer ones, all of the impressions having been made on the same machine by the same operator and using the same quality of paper.

After rating the original writing as above, strips of the same were exposed to sunlight for ten days and to the various wet reagents for one hour, dried and again rated according to the following scale:

1. Unaffected.
2. Very slightly affected, just distinguishable.
3. Slightly affected.
4. More than 3.
5. Much affected.
6. Still more than 5.
7. Very badly affected.
8. Almost effaced, letters distinct.
9. Almost effaced, letters indistinct.
10. Completely effaced.

The method of ranking may not be clear from the mere lists above, but the actual scales, composed of writing classified as the scales indicate, made it possible to rank the samples with comparative ease. In the case of copy papers, strips of the original writing, of the writing that had served for copy and the copy itself were exposed to the same tests and separately ranked. In Table III the three figures appearing in each column for copy papers give the rating of original, copied writing and copy in the order named.

For the sunlight test, the exposure was made under glass in a picture frame, and the number of days recorded included only those of direct sunlight. The following wet reagents were used:

1. Water at room temperature.
2. 0.5 per cent. HCl in water.
3. 0.5 per cent. HCl in alcohol.

4. 2 per cent. ammonia in water.
5. 2 per cent. ammonia in alcohol.
6. N/200 calcium hypochlorite.

Very delicate reagents were found to give better comparative results, and for the same reason the time of exposure was limited to one hour. Strips of the paper in Erlenmeyer flasks were covered with the reagent and the immediate effect as well as that after each fifteen minutes was noted. Only the final result after an hour's action is recorded in the table. It is noteworthy that water, which with the exception of the copy papers does not affect the ink as a solvent, does remove it mechanically to a material extent, and with the other reagents also the largest part of their effect is due to the displacement of the ink bodily, and not to any chemical or solvent action.

* * * * *

Beginning with Table III the papers which show the best writing qualities and the most resistant ink are numbers 2288, 2289, 2291*a*, 2291*b*, 2300 and 2303 among the black record class; numbers 2302 and 2382 of the blue record; number 2291*c* among the copy papers and possibly number 2301 of the purple record. Numbers 2288, 2289, 2300 and 2303 from Table I have the best paper backing but 2303 does not give a good stretch test and from Table II has but 44 per cent. of ink, indicating that it would be short-lived. Number 2288 has also a small quantity of ink. The choice lies then between numbers 2289 and 2300, made by different manufacturers of established high standing, number 2300 having the advantage of being a feather weight paper with exceptionally high tensile strength and stretch.

The purple record papers were made by the same firm and are of nearly equal quality, the advantage being slightly in favor of 2301 in the tests of the writing and the strength of the paper foundation, and with the disadvantage of having somewhat less ink. Of the blue record papers, number 2382 though of lighter weight has the greater strength and much more available ink. Of the copy papers, number 2291*c* has very high strength and stretch but unfortunately only a small amount of ink. For some of the tests of writing number 2306 stands higher than 2291*c* and the strength of the paper is greater, but the amount of ink is possibly too low to give long service.

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GLUTEN FEEDS—ARTIFICIALLY COLORED.

BY EDWARD GUDEMAN.

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Gluten feeds are the by-products obtained in the starch, glucose, corn sirup and starch-sugar industries. In the process of manufacture the