and sufficient nitric acid to convert all the lead present to lead nitrate. Should there be any separation of basic lead nitrate add more water until complete solution is effected.

In the analysis of red lead, treat one gram of the sample with 10 cc. of water and sufficient nitric acid to convert all the oxide present, excepting the lead dioxide, to lead nitrate. Add 10 cc. of hydrogen peroxide (1 : 3.5) to the solution, and boil for a minute until all the oxides of lead pass into solution.

After complete solution of either the metallic lead, litharge or red lead, boil the nitrate solution gently for several minutes to convert all the iron to the ferric condition, cool the contents of the beaker, neutralize the free nitric acid present with ammonium hydroxide and render faintly acid with nitric acid. A solution made from red lead may, after neutralization and the subsequent addition of acid, show the presence of lead dioxide. When this occurs, the solution can be rendered clear by the addition of a few drops of the hydrogen peroxide solution.

Wash the contents of the beaker into a 100 cc. Nessler cylinder, add 15 cc. of dilute ammonium sulphocyanide (1 : 20) and dilute to the mark. The intensity of the blood-red color depends upon the amount of iron present. The color is compared with a blank made in the following manner:

A solution of ferric ammonium sulphate of known strength is required. This is made by dissolving 0.7022 gram of ferrous ammonium sulphate in water. Acidify with sulphuric acid, heat to boiling and add a solution of potassium permanganate until all the iron is converted to the ferric condition. Only the very slightest pink tinge may be present after the addition of the potassium permanganate, as this tinge will fade away, while the presence of a pink color tends to vitiate the results. Allow the solution to cool and dilute to one liter. One cc. of this solution equals 0.0001 gram of iron.

Prepare the blank by pouring into a 100 cc. Nessler cylinder 10 cc. of hydrogen peroxide of the above strength, provided hydrogen peroxide was added for the solution of the oxide, approximately the same amount of nitric acid as was required for the solution of the original metallic lead or oxide, and 15 cc. of ammonium sulphocyanide solution. Dilute to 100 cc. and titrate to the exact color developed in the sample under examination, by the addition of the standard ferric ammonium sulphate solution. One cc. of this solution equals 0.01 per cent. of iron when a one-gram sample is used. The percentage of iron present should not require more than 2 or 3 cc. of the standard to equal it, or the color will be too deep for comparison.

It will be found that the color can be accurately compared to within 0.001 per cent. of iron content. The small percentage of copper present in lead and its oxides does not interfere with the reaction.

LABORATORY OF FICHER LEAD COMPANY,
JOPLIN, MO.

A RAPID MICROSCOPICAL METHOD FOR THE DETERMINATION OF ARSENIC, AS ORPIMENT, IN SHELLAC.¹

By ROBERT SCHWARZ.

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As is well known, orpiment, the mineral form of arsenic trisulphid, is very generally added to shellac in India, where this product is collected and prepared for the market. Orpiment is added merely to make shellac more opaque and light straw-colored.

An attempt has therefore been made to obtain from India shellac to which no orpiment has been added, but it has been found that, for some unknown reason, almost all shipments of so-called "arsenic-free" goods contain from small amounts to considerable quantities of arsenic, mostly in the form of orpiment. Therefore it appeared advisable to develop a method of qualitatively determining orpiment in shellac, which method would be more rapid than the Gutzeit or Marsh tests. Such a method has been evolved and bases itself, (1) upon the fact that orpiment is insoluble in alcohol and can be precipitated from alcoholic solution of shellac by centrifugal motion, and (2), upon the characteristic appearance of orpiment under the microscope. When examined in this manner, orpiment appears as transparent to translucent granular masses and imperfect crystals of a distinctly characteristic yellow-greenish color.

The method, therefore, is very simple and rapid, as can be seen from the following:

A 10-gram average sample is ground fine in a coffee mill and then dissolved in 15 cc. of methyl alcohol, solution being hastened by shaking and warming the flask. The varnish is then poured into a graduated tapering tube and this revolved in a centrifugal machine for 5-6 minutes at 1000-1500 revolutions per minute. This causes the precipitation of the greater part of the insoluble material, which contains the orpiment. The varnish is then decanted, the precipitate shaken with methyl alcohol and the tube again revolved in the centrifuge for one to two minutes. The alcohol is then decanted as thoroughly as possible, the amount of the precipitate recorded, and a small portion transferred to a microscopic slide by means of a capillary tube. This preparation is then carefully examined under the microscope, using a magnification of 450-600 diameters.

With an ordinary brewers' shellac or varnish containing a normal amount of insoluble material and above 150 parts of arsenic per million, several pieces of orpiment will be observed in each field. Some shellacs, however, contain a considerable amount of insoluble material, and so the precipitate becomes larger and the number of pieces of orpiment in each field correspondingly smaller. The method has been used not only on the common grades of shellac, but it has also been tried on so-called "arsenic-free" shipments and three bleached shellacs. The results of all tests have been embodied in a table in which is given the number of each test, description of the sample, amount of the precipitate, number of pieces of orpiment

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found in each field or preparation, and the amount of arsenic trioxide, as determined by the Marsh or Gutzeit test. The Marsh or Gutzeit test determines the total amount of arsenic present, and therefore, as Langmuir and White¹ have shown that a certain amount of orpiment may be dissolved in the shellac in the process of manufacture, it is not surprising that in some instances the microscopic tests failed to reveal orpiment, while the chemical tests showed appreciable amounts of arsenic to be present.

Sample Number.	Description.	Sedi- ment.	Orpiment crystals per field.	AS ₂ O ₃ . Parts per million.	
				M=Marsh	G=Gutzeit.
1 Varnish	Orange	0.1 cc.	4-5	400 M	
2 Varnish	Orange	0.05	1	60 M	
3 Varnish	Orange	0.05	4-5	155 M	
4 Varnish	Orange As-free	0.025	none ¹	none G	
5 Varnish	Manheim	none	none	none M	
6 Varnish	Manheim	0.02	1-2	112 M	
7 Varnish	White	0.05	none ¹	none G	
8 Shellac	Grain Lac.	1.5	1	45 M	
9 Shellac	R & P garnet	0.1	none ¹	85 M	
10 Shellac	Gr. orange	0.075	none ¹	none M	
11 Shellac	Reg. orange	0.075	6-8	550 M	
12 Com. As-free shellac		0.025	1 ²	4 G	
13 Com. As-free shellac		0.05	1 ²	7.5 G	
14 Com. As-free shellac		0.025	2 ²	4 G	
15 Com. As-free shellac	Grain lac.	1.00	1 ²	20 G	
16 Com. As-free shellac	Grain lac.	1.00	1 ²	20 G	
17 Bleached shellac		0.01	1 ²	6 G	
18 Bleached shellac		0.25	1 ³	traces G-M	
19 Bleached shellac		0.01	none ¹	15 G	

¹ Three preparations examined.

² Number of crystals in each preparation.

³ One crystal in every third preparation.

It is easily seen from the above table that the sediment from a brewers' varnish (samples 1-6), which is generally made from the ordinary grades of shellac, contains such an amount of orpiment that there are always one or more crystals present in each microscopic field. Sample 4, however, did not show any orpiment in three microscopic preparations, and as experience had shown that this number of preparations would show at least one crystal of orpiment, if any were present, this sample was judged orpiment-free. The Gutzeit test showed that no arsenic in any form was present.

Of the raw or purified shellacs all but two showed crystals of orpiment where arsenic was subsequently found by the Marsh or Gutzeit tests. The two exceptions were No. 9, of which a number of preparations were made and no orpiment found, and No. 19, a bleached, wax-free sample, which gave similar results. It is quite probable that in both instances the arsenic was in solution, and that any orpiment which had been present in the raw lac had been removed in the process of manufacture.

Regarding samples Nos. 12-18, it is interesting to note that the method will show the presence of orpiment even when the total arsenic present is only 4 parts per million, and in one instance—sample No. 18—one crystal was found in every third microscopic preparation, while the Gutzeit and Marsh tests showed the presence of traces only of arsenic.

As regards using the method to obtain quantitative approximation of the amount of orpiment present, it

must be stated that, in the first place, the method is suggested only as a qualitative test. However, by using the same microscope, ocular and objective, and by comparing the sediments of unknown shellacs with those of samples containing known amounts of orpiment, a fair idea of the amount of orpiment present can be obtained.

In conclusion I wish to acknowledge my indebtedness to Dr. A. C. Langmuir, at whose suggestion this paper was written, and who has given valuable advice in the preparation of the same.

MICROSCOPICAL LABORATORY,
FIRST SCIENTIFIC STATION FOR THE ART OF BREWING,
NEW YORK CITY.

SOME PRACTICAL METHODS FOR TESTING AND IDENTIFYING COLORS FOR PRINTING INKS AND SIMILAR PIGMENTS.

By SAMUEL LEVINSON.

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Every manufacturer, in fact every large user of colors, should be in position to identify and match colors brought to his attention as a producer, jobber or buyer. In this paper we shall consider only the so-called LAKE COLORS, *i. e.*, colors made from coal-tar dyestuffs consisting of the base, coloring matter and usually some substance or substances used to fasten the dye to the base. Analytical methods very often are insufficient to properly identify chemical preparations. A complete analysis giving the percentage of all the constituents which can be properly identified chemically will often be of comparatively little value. But on the other hand a synthetic test, *i. e.*, a combination of different materials which results in a substance identical with the one tested will make the examination complete and at the same time provide all the important analytical data. In very many cases the synthetic test is simpler and takes up much less time, than an analysis. In many factories where there are no or very limited laboratory facilities a complete analysis is out of the question and one must per force take refuge in synthetic methods. Certain conditions, of course, cannot be duplicated in the laboratory and besides the chemist must be familiar not only with analytical methods but must understand the manufacture of the articles in question. Happily in the color line conditions are such that almost any color can easily be prepared in the laboratory and the preparation requires but little time and very limited facilities.

It is relatively easy to find out the percentage of coloring matter in a color and in many instances the manufacturer will state the nature of the dye used, but, if the consumer should pay according to the percentage of dye, as revealed by such analysis, the results might prove disastrous. It is quite a common occurrence for two colors with exactly the same percentage of dye to show different strengths. In examining a new color it is worth while to remember that there are relatively few lake colors in the market in shades other than RED, because some of the more important INORGANIC colors, like chrome yellow, chrome green, ultramarine and prussian blue, are