so that the chances of them occurring in the liquid condition are small. No account is taken herein of gases that are closely associated under heavy pressure with oil in the sands. Under such condition there is a solution of gases in the oil to a great extent. The natural gas used in Pittsburgh is not associated with oil in the earth's strata.

CHEMICAL LABORATORY
BUREAU OF MINES, PITTSBURGH

THE ANALYSIS OF CHROME YELLOWS AND GREENS

By A. GIVEN

Received January 22, 1915

From inquiries received at this laboratory, it appears that the determination of the composition of chrome yellows and chrome greens is not well understood in many color and paint works; and the excellent publications of the Bureau of Standards on the analysis of inks help only in a degree. The method here given is the result of a great deal of work and experience with these colors, and has given excellent satisfaction.

METHOD

MOISTURE AND LEAD CARBONATE—Weigh out accurately about 1 gram of the yellow or green into a small beaker, and dry for 4 hours at 105–110°C. Calculate the loss in weight as water. Add 50 cc. of 50 per cent acetic acid to the dry substance in the beaker, mix thoroughly, and let stand over night. Filter through a tared Gooch, wash with hot water, dry, cool and weigh. Calculate the loss from dry weight as lead carbonate.

LEAD SULFATE AND CHROMATE—Weigh out accurately about 1 g., wash into a 250 cc. beaker, add 50 cc. water and 50 cc. of 25 per cent caustic soda and boil for 5 or 10 minutes. Filter through a tared Gooch, wash thoroughly with hot water, cool the filtrate and make up to 250 cc. Save the residue. To 50 cc. of the solution add an excess of hydrochloric acid and 5 cc. alcohol and boil until the chromate is reduced to chromic chloride. Add an excess of barium chloride, boil, let settle, filter on an ashless paper, wash, burn and weigh as barium sulfate. Calculate to per cent lead sulfate.

To 50 cc. of the above solution add an excess of nitric acid, heat to boiling and add 25 cc. boiling saturated solution of potassium bichromate. Boil 1 minute, let settle, filter through a tared Gooch, wash, dry and weigh. Calculate as per cent lead chromate. From this amount subtract the lead sulfate and lead carbonate, both calculated to lead chromate, and call the difference actual lead chromate present.

RESIDUE—Fill the Gooch containing the residue from the solution for lead sulfate and chromate with hot 1 : 1 hydrochloric acid, let stand 10 minutes, filter and wash with hot water. Repeat twice, wash thoroughly, dry and weigh. Calculate remaining residue as per cent barytes or china clay according to base.

PURE BLUE—The sum of all the previous determinations is subtracted from 100, and the difference called pure blue.

LABORATORIES OF MORRIS HERRMANN AND COMPANY
878 Mt. Prospect Avenue, Newark, New Jersey

THE UTILITY OF SULFUROUS ACID AND PURE YEAST IN CIDER VINEGAR MANUFACTURE

By W. V. CRUESS, J. R. ZION AND A. V. SIFREDI

Received January 4, 1915

A study of methods of alcoholic fermentation of apple juice destined to be made into vinegar in various California factories showed that in almost every case no attempt was made to control the microorganisms of fermentation. A brief description of the various ways of handling the cider stock will no doubt be of interest.

In one of the largest factories the cull apples and peels and cores from apple driers are ground or "grated" in an Ohio apple grater as soon as received. The juice is pressed out with heavy screw presses driven by a motor and cog gearing with a capacity of 60 tons per day. The juice goes to 20,000 gallon tanks. The first tank of the season is started with a large starter of compressed yeast. When this tank is in fermentation, about one-third of its contents is used to start the next tank. The third tank is started from the second, and so on through the whole series of tanks. Over 500,000 gallons are made in this way during a season. Examination of the fermented juice showed it to be "dry;" that is, practically free from unfermented sugar, but it was shown by microscopical examination to contain large numbers of lactic acid bacteria and the large tanks soon developed a heavy growth of *mycoderma* (wine flowers) after alcoholic fermentation. The lactic bacteria develop a "mousey" flavor and the *mycoderma* rapidly oxidizes alcohol to CO₂ and H₂O without forming any corresponding amount of acetic acid.¹ Laboratory tests demonstrated that *mycoderma* isolated from cider was capable of destroying all of the alcohol of a fermented orange juice containing 4.5 per cent alcohol in three weeks. The gravity of heavy *mycoderma* growth may be seen from these figures.

Another large factory stores its cull apples, peels and cores in a large wooden bin. Often this material undergoes a fermentation, resembling silage fermentation, before it can be crushed. The juice after crushing and pressing is allowed to undergo spontaneous fermentation in 30,000-gallon tanks. Wild yeasts, lactic bacteria and *mycoderma* develop profusely, giving a very cloudy cider. Occasionally a tank "sticks" with some unfermented sugar, but in general the fermentations are complete.

A third factory (until 1914) crushed its apples and allowed the crushed apples to undergo spontaneous fermentation 3 or 4 days before pressing. This method gave a high yield of juice, due to the softening effect of the fermentation. Beginning with this season, this factory used pure "Burgundy" wine yeast on the crushed apples with good results. The yeast was propagated according to *Bulletin 230* of the University of California Experiment Station. No sulfurous acid is used on the crushed apples, consequently the fermented cider shows a strong tendency to develop *mycoderma*.

The above factories all use the generator process

¹ A. V. Sifredi, Thesis for M. S., Univ. Calif., Dec., 1914.

for converting the alcohol to acetic acid. A fourth factory making 50,000 to 75,000 gallons per year uses the slow process throughout. Fermentation is allowed to take place spontaneously with the result that lactic acid bacteria, as well as *mycoderma*, grow vigorously in the fermented cider. There are also a few fermentations that "stick," *i. e.*, stop before the sugar is completely transformed to alcohol.

On account of these obvious defects in present methods, the experiments described below were carried out to see if the beneficial effects of pure yeast

was added. It took three days to fill the 30,000-gallon tank. By the time the tank was full in each case, fermentation had progressed so far that no attempt at a fermentation record was made. Table II shows the effects due to the sulfurous acid and pure yeast: gain due to pure yeast and SO₂ was found by subtracting 15.20 from 16.27 to give difference in sugar = 1.07 per cent. This was divided by 1.7 to get its alcohol volume per cent equivalent as it is considered that 1.7 per cent sugar gives 1 per cent alcohol in practice. This figure, 0.73 per cent, subtracted from 2.05 gives

TABLE I—RESULTS OF EXPERIMENTS WITH SULFUROUS ACID AND PURE YEAST IN BARREL TESTS

FERMENTATION RECORD BARREL A (NATURAL FERMENTATION)			FERMENTATION RECORD BARREL B (PURE YEAST AND SO ₂)			ANALYSES OF BARRELS A AND B Total reducing				
Date	Balling	OBSERVATIONS	Date	Balling	OBSERVATIONS	SAMPLE	Ball- ing (Brix)	Total acid as acetic	matter sugar	Alcohol per cent by vol.
Sept. 5	16.0	Filled barrel A	Sept. 5	16	Filled barrel B and added SO ₂	Original juice	16.0	0.60	15.00	0.00
Sept. 8	13.5	Fermenting vigorously	Sept. 6	16	Racked into another barrel and added 2 per cent by volume of pure Burgundy yeast	Barrel A Jan. 9	0.0	0.84	0.55	6.20
Sept. 13	5.0	Fermenting slowly	Sept. 8	14	Fermenting vigorously; taste clean; aroma much cleaner than "A"	Barrel B Jan. 9	0.0	0.61	0.037	8.00
Sept. 19	3.0	Fermenting very slowly	Sept. 30	1	Fermenting slowly; microscope shows absence of wild yeasts or bacteria of any kind	Barrel A Apr. 5	...	1.18	0.60	5.60
Sept. 30	3.0	Strong "mousey" flavor: many lactic bacteria Still tastes sweet	Jan. 9	0	Taste clean and dry. Much clearer than "A"	Barrel B Apr. 5	...	5.90	...	2.56
Oct. 13	3.0									
Jan. 9	0.0									

and sulfurous acid obtained with wine fermentation in 1911¹ could be duplicated with apple juice fermentation.

SMALL SCALE FERMENTATIONS

Two barrels of the same juice were taken. Barrel "A" was allowed to ferment naturally; barrel "B" was treated with potassium metabisulfite (K₂S₂O₅) at the rate of 12 oz. per 200 gallons or approximately 175 mg. SO₂ per liter. It was allowed to stand 24 hours; was then drawn off into another barrel and pure "Burgundy" wine yeast was added. A summary of the two fermentations appears in Table I and may be stated as follows:

- 1—Increase of 1.8 per cent alcohol.
- 2—More complete fermentation of sugar.
- 3—Elimination of wild yeasts and lactic bacteria; probably due to action of SO₂ during first 24 hours.
- 4—More rapid clearing of the cider and the vinegar.
- 5—Improved and cleaner flavor.
- 6—More rapid acetification after alcoholic fermentation.

30,000-GALLON FERMENTATIONS

A 30,000-gallon tank (No. 10—Table II) of apple juice was filled with juice from culls, peels and cores during a pressing period of three days. Samples were taken during filling and analyzed for Balling degree, alcohol and total acid. The juice was allowed to ferment spontaneously and was handled by methods ordinarily used in this factory.

A similar 30,000-gallon tank (No. 16) was filled with similar juice. Before going to the large tank, each 1,000 gallons as it came from the press was held temporarily in a 1,000-gallon vat and treated with 8 oz. metabisulfite per 200 gallons. This was added from a solution of 8 oz. metabisulfite per gallon in water. The first 2,000 gallons to go into the large tank were started with 100 gallons pure "Burgundy" yeast and left till next morning before any more juice

1.32, the "net increase" in alcohol due to pure yeast.

The results of the large scale fermentation were satisfactory, showing a very material increase in al-

TABLE II—RESULTS OF 30,000-GALLON TANK TESTS
Results in Percentages

	Tank No. 10. Natural fermentation	Tank No. 16. Pure yeast and SO ₂
BEFORE FERMENTATION:		
Total solids + (alcohol × 1.7). Average of samples.....	15.20	16.27
AFTER FERMENTATION:		
Total acid as acetic.....	0.568	0.652
Volatile acid as acetic.....	0.160	0.108
Extract.....	3.70	3.60
Alcohol: Volume per cent.....	6.35	8.40
CALCULATIONS:		
Difference in total solids + (alcohol × 1.7) in tanks 10 and 16.....		1.07
Alcohol equivalent to difference in total solids.....		0.73
Difference in alcohol in fermented liquids.....		2.05
Net gain in alcohol due to pure yeast and SO ₂		1.32

cohol due to pure yeast and sulfurous acid. Microscopical examination showed that the pure yeast and sulfurous acid gave a much cleaner fermentation than the natural fermentation.

SUMMARY

The use of pure yeast and sulfurous acid in barrel fermentations gave increased yield of alcohol, more complete fermentation of the sugar, a better flavor, more rapid clearing, a fermented liquid practically free from lactic bacteria and *mycoderma vini*, and a more rapid change of alcohol to acetic acid after alcoholic fermentation. This indicates disappearance of practically all of the active sulfur dioxide because acetic bacteria have been shown to be very sensitive to it. Large scale fermentations with pure yeast and sulfurous acid gave an increased yield of alcohol and a cleaner fermentation. Examination of several factories shows the extreme need of some method of control of alcoholic fermentation, and to supply this need the use of small amounts of sulfurous acid to eliminate wild yeast and the addition of pure selected yeast to give a rapid and complete fermentation seems the most practical means of producing the desired results.