

consistency; at any rate, we have no positive knowledge that the so-called fixed acids occurring in the final residue represent the actual fixed acids in the original wine. A titration of the residue may suffice as an indication of the acids present after driving off the volatile constituents by the prolonged heating, but to employ the result of such a titration as a factor in the calculation of the actual volatile acids appears to be an unwarrantable proceeding.

In expressing the results of analysis, the orthodox custom appears to be to calculate the fixed and total acids as tartaric and the volatile acids as acetic. It is impossible to concede any advantages in favor of this custom. It may be safe to assume that in wines the fixed acids are in the main tartaric and the volatile acids acetic; but, even on such assumptions, the results are strictly erroneous and not readily comprehensible. Such a method applied to the various fruit juices and ciders would fail to give significant results in practically all cases, and the case is still worse when one undertakes to apply the method of calculating the acids as sulphuric. Instead of these conventional methods, it has been found better to adopt the plan of expressing all results for total, fixed and volatile acids in terms of the number of cc. of normal acid in a definite measure, say 100 cc., of wine. There will then be afforded results which can be readily compared and comprehended. Furthermore, in case it be required to calculate results in terms of any particular acid, such an operation can easily be carried out.

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A COMPARISON OF METHODS FOR THE PREPARATION OF MILK SERUM.

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It is a well-known fact that milk serum is of more uniform composition than milk, consequently its constants, particularly the specific gravity have been used for many years for the detection of added water. Recently the index of refraction has been suggested for this purpose and was first employed by Villiers and Bertault¹ in 1898 who prepared the serum by placing a mixture of two volumes of 1 per cent. acetic acid and one volume

of milk in a flask connected with a reflux condenser, heating to the boiling point, cooling and filtering. The serum was examined in the oleo-refractometer of Amagat and Jean. Matthes and Müller¹ in 1903 first suggested the use of the Zeiss immersion refractometer for this purpose, using the serum obtained from spontaneously soured milk. Leach and Lythgoe² in 1904 prepared the serum according to the method of Woodman³ with 25 per cent. acetic acid, taking the refraction with the immersion refractometer. Baier and Neumann⁴ mixed the milk with an asapol citric acid solution in the cold, filtered, and determined the index of refraction of the filtrate with the Wollny milk fat refractometer. Ackerman⁵ heated the milk with a calcium chloride solution and determined the index of refraction of the serum by means of the immersion refractometer.

During the past two years many samples of milk of known purity have been examined in the laboratory of food and drug inspection of the Mass. State Board of Health to which two or more of the above methods have been applied. The specific gravities of the sera were obtained at 15°, and the indexes of refraction on the immersion refractometer at 20°, and the results are reported in the accompanying tables. The samples of milk were all milked in the presence of an inspector or an analyst of this department.

The details of the methods used for the preparation of the milk sera are as follows:

Acetic Acid Method.—To 100 cc. of milk at about 20° C. add 2 cc. of 25 per cent. acetic acid, mix well and place in a water bath at 70° C. for 20 minutes. Cool 10 minutes in ice water and filter.

Natural Souring Method.—Allow the samples to sour spontaneously and filter off the serum.

Calcium Chloride Method.—The details have been slightly modified from the method of Ackerman indicated above. Place 90 cc. of milk in a flask, add 0.75 cc. of calcium chloride solution, sp. gr. 1.1375 (when diluted 1:10 this solution reads 26 on the immersion refractometer at 17.6° C.), shake thoroughly, close the flask with a cork carrying a glass tube to act as a reflux condenser, place in a boiling water bath for 20 minutes, cool to 20°, mix the condensed water and serum without shaking, and filter.

¹ *Zeit. offenl. Chem.*, **10**, 173.

² *J. Am. Chem. Soc.*, **26**, 1195.

³ *Ibid.*, **21**, 503.

⁴ *Zeit. Nahr-Genussm.*, **13**, 369.

⁵ *Ibid.*, **14**, 186.

¹ *Bull. Soc. Chim.*, **19**, 305.

Asaprol Method.—The precipitating solution is made by dissolving 30 grams of asaprol and 55.8 grams of crystallized citric acid in 1 liter of water. If the refraction is not 36.3 at 20° C. on the scale of the immersion refractometer add citric acid or water to make it so. Mix equal volumes of the above solution and the milk, shake well and filter.

TABLE I.
REFRACTION OF MILK SERA FROM KNOWN PURITY MILK.

<i>Individual Cows.</i>			
Method.			
Acetic acid.	Natural souring.	Calcium chloride.	Asaprol.
46.2	47.7
45.9	44.6	..	38.4
45.8	44.0
45.7	43.4	40.1	37.0
45.6	43.5
45.5	41.5	38.7	37.1
45.1	41.5	39.0	36.8
44.9	41.5	39.8	36.1
44.8	43.8
44.8	43.7	39.2	36.0
44.7	43.5	..	36.6
44.6	..	38.5	36.6
44.5	45.0
44.3	42.8	..	35.7
44.4	43.8	..	36.7
44.3	43.0
44.3	42.8
44.3	42.3	39.0	37.0
44.2	41.2	38.6	37.0
44.2	41.0
44.1	43.0	39.1	36.7
44.1	40.7	38.2	36.8
44.0	42.2	38.7	..
43.9	44.5
43.9	42.6
43.8	44.0
43.8	44.0
43.8	43.0	..	36.7
43.8	41.6
43.7	44.2	..	37.5
43.7	42.6
43.7	42.4
43.7	41.5	..	36.9
43.6	43.0
43.6	43.0	40.0	36.3
43.6	42.0	38.6	36.3
43.5	43.5
43.5	42.8
43.5	41.0	38.4	36.3
43.4	43.1
43.2	43.3
43.2	42.2
43.2	41.8	38.9	36.6
43.2	40.9	..	37.4
43.0	43.7
43.0	43.2
43.0	42.8
43.0	42.3
43.0	42.0
43.0	41.7
43.0	41.5
43.0	41.4	39.1	36.4
43.0	41.1	38.4	36.6
43.0	40.9
42.9	43.6	..	36.5
42.9	43.0
42.9	41.4	39.0	36.7
42.8	37.4
42.7	..	38.8	..
42.7	..	38.0	..

TABLE I—(Continued).

Method.			
Acetic acid.	Natural souring.	Calcium chloride.	Asaprol.
42.6	41.3	38.1	36.1
42.5	42.2	39.3	36.3
42.5	41.5
42.5	..	38.2	36.6
42.4	43.3	..	36.4
42.3	43.7	39.0	37.0
42.3	41.9	..	36.8
42.3	41.6	39.8	36.6
42.3	40.8	38.8	36.7
42.2	42.0	..	37.0
42.2	41.0
42.1	44.0	..	36.8
42.1	43.7	..	36.6
42.0	41.0
42.0	40.3	37.1	36.2
42.0	40.2	36.8	35.8
41.8	40.5
41.7	40.9	38.2	36.1
41.7	..	38.0	..
41.7	40.0	36.8	36.2
41.6	43.9	..	36.0
41.5	40.4	36.4	35.7
41.4	40.3	38.4	35.6
41.3	40.3
41.2	40.0
40.6	40.7	36.6	35.8
40.5	39.3
40.4	38.3
40.0	40.1
Mixed Milk of Known Purity.			
43.6	42.9	39.0	37.5
43.5	42.0	38.7	..
43.4	40.8	38.2	36.7
42.5	41.0	39.4	36.3
42.1	41.3

TABLE II.
SPECIFIC GRAVITY OF MILK SERA FROM KNOWN PURITY MILK.

<i>Individual Cows.</i>			
Method.			
Acetic acid.	Natural souring.	Calcium chloride.	Asaprol.
1.0333	1.0259
1.0330	1.0324
1.0329	1.0291
1.0327	1.0297	1.0275	1.0256
1.0324	1.0263	1.0274	1.0251
1.0323	1.0295	1.0276	1.0253
1.0322	1.0278	1.0272	1.0253
1.0322	1.0272	1.0274	1.0252
1.0320	1.0290
1.0319	1.0284	1.0270	1.0251
1.0318	1.0245
1.0318	1.0290	1.0254
1.0318	1.0260
1.0318	1.0273	1.0264	1.0252
1.0316	1.0275	1.0263	1.0253
1.0316	1.0300	1.0256
1.0316	1.0292	1.0252
1.0316	1.0300
1.0314	1.0281	1.0267	1.0252
1.0314	1.0306
1.0313	1.0276	1.0274	1.0251
1.0313	1.0282	1.0265
1.0312	1.0287	1.0268	1.0252
1.0312	1.0280	1.0269	1.0249
1.0312	1.0272	1.0250
1.0312	1.0282	1.0268	1.0252
1.0312	1.0285	1.0264	1.0244
1.0312	1.0295
1.0311	1.0235	1.0264	1.0250
1.0311	1.0272	1.0259	1.0249
1.0310	1.0277	1.0274	1.0248
1.0310	1.0236

TABLE II—(Continued).

Acetic acid.	Method.		Asaprol.
	Natural souring.	Calcium chloride.	
1.0310	1.0275
1.0310	1.0270
1.0310	1.0284
1.0309	1.0270	1.0265
1.0309	1.0310	1.0244
1.0308	1.0290	1.0247
1.0308	1.0295	1.0247
1.0308	1.0253
1.0308	1.0272
1.0307	1.0248	1.0270	1.0252
1.0307	1.0267	1.0274	1.0248
1.0307	1.0278
1.0306	1.0248
1.0306	1.0295	1.0256
1.0306	1.0304
1.0305	1.0292
1.0305	1.0306
1.0305	1.0274	1.0259	1.0245
1.0304	1.0245
1.0304	1.0285
1.0304	1.0270
1.0303	1.0260	1.0266	1.0252
1.0303	1.0294
1.0302	1.0265	1.0261	1.0247
1.0302	1.0282	1.0271	1.0249
1.0302	1.0290	1.0285	1.0243
1.0302	1.0285	1.0246
1.0302	1.0255
1.0302	1.0273	1.0256	1.0247
1.0301	1.0292
1.0301	1.0272	1.0258	1.0245
1.0301	1.0206	1.0264
1.0300	1.0290	1.0263	1.0245
1.0300	1.0293	1.0246
1.0300	1.0280	1.0245
1.0300	1.0293
1.0300	1.0264
1.0299	1.0280	1.0242
1.0299	1.0283
1.0299	1.0296
1.0298	1.0229	1.0258	1.0246
1.0298	1.0255
1.0298	1.0260
1.0298	1.0285
1.0298	1.0306
1.0296	1.0266
1.0296	1.0279
1.0296	1.0282
1.0296	1.0253	1.0259
1.0295	1.0264
1.0295	1.0279
1.0295	1.0306
1.0294	1.0274
1.0293	1.0282
1.0293	1.0223	1.0258
1.0292	1.0290	1.0235
1.0292	1.0285
1.0292	1.0270	1.0248	1.0244
1.0291	1.0276
1.0290	1.0236	1.0268	1.0238
1.0290	1.0264
1.0290	1.0268	1.0244	1.0240
1.0289	1.0261	1.0252	1.0239
1.0288	1.0221	1.0248	1.0241
1.0287	1.0307
1.0282	1.0249
1.0280	1.0350
1.0280	1.0254	1.0237
1.0280	1.0272
1.0280	1.0262	1.0234	1.0241
1.0274	1.0266
Mixed Milk of Known Purity.			
1.0312	1.0292	1.0264
1.0310	1.0284

TABLE II—(Continued).

Acetic acid.	Method.		Asaprol.
	Natural souring	Calcium chloride.	
1.0308	1.0266
1.0306	1.0283	1.0258	1.0251
1.0302	1.0249
1.0300	1.0284

Of the above methods, the asaprol method is the easiest of manipulation, gives the clearest serum in the least time and shows the lowest refraction with the least variation. Unfortunately pure asaprol is very difficult to obtain and owing to the fact that it decomposes readily, it is not an easy matter to prepare different solutions that will give identical sera with the same sample of milk.

The calcium chloride method is the most difficult of manipulation and is liable to give a cloudy serum rather troublesome to read but the figures do not vary so much and are lower than those obtained by the acetic acid method. The natural souring method is too slow for ordinary use, but is valuable in the hot weather if the milk is nearly sour by the time it reaches the analyst. As a rule, the figures are lower than those obtained by the acetic acid method, but in a few cases they run higher. Four years' experience with the acetic acid method has shown it to be reliable, easy of manipulation, and to give concordant results. It is still used in this laboratory for the detection of added water in milk.

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NOTES.

ON THE USE OF INCANDESCENT LAMPS WITH VOLATILE SOLVENTS.

For the extraction with, and distillation of, volatile solvents the ordinary 8- and 16-candle power incandescent lamps may be employed to advantage as a source of heat. The 8-candle power lamp has been found to be more satisfactory for (ether) extraction, and the 16- for distillation. The lamp as used is enclosed in a slightly tapering cone, or cylinder of asbestos paper. By placing asbestos paper around lower part of extraction apparatus the flow of condensed solvent is easily regulated, and once regulated requires no further attention. Asbestos paper will also be found useful if placed around flask during distillation. Incandescent lamps may also be used to advantage for evaporation at low temperatures, especially when inflammable vapors are given off, the temperature being regulated by raising or lowering the source of heat.

The use of electricity for boiling the solvent during extraction gives a compact apparatus, everything being on