

EXPERIMENT 1.

Cell	Amp.	Volts	C. D. Anode amps. per sq. cm.	C. D. Cathode amps. per sq. cm.	Cathode
1	3.6	4.5	0.06	0.03	Graphite
2	3.6	6.0	0.06	0.17	Magnesium
3	3.6	6.0	0.06	0.17	"
Amp. Hrs.	Theoretical Cl. Chlorine grams	Chlorine made grams	Chlorine per liter	Efficiency	
60	79	50.7	26.6	64%	
Loss of Magnesium					
Weight before	grams	.....	Cell w 2	Cell 3	
"	after	"	0.7	0.7	
"	"	"	0.62	0.675	
Loss	.....	0.08	11.5%	0.025	3.6%

EXPERIMENT 2.

Cell	Amp.	Volts	C. D. Anode amps. per sq. cm.	C. D. Cathode amps. per sq. cm.	Cathode
1	4.5	5.5	0.12	0.24	Graphite
2	4.0	6.0	0.064	0.19	Magnesium
3	4.5	6.5	0.074	0.21	"
Amp. Hrs.	Theoretical Chlorine grams	Chlorine made grams	Chlorine per liter	Efficiency	
62	82.4	48.2	25.4	58.5%	
Loss of Magnesium					
Weight of wire before	.....	Cell 2	Cell 3		
"	"	"	0.75	0.75	
"	"	"	0.675	0.724	
Loss and per cent	.....	0.075	10%	0.026	3%

Power required for 100 lbs. chlorine is 450 H. P. hours.

Loss of magnesium for 100 lbs. chlorine is 1/2 lb. or less.

We also tried the addition of traces of caustic soda to the salt solutions, before electrolysis, and found this had a beneficial action in reducing the loss of magnesium from the cathode.

The great advantage in the use of the magnesium cathode would seem to be not only the lower power cost, but that a large quantity of the salt can be converted, it being possible to convert from 35 to 50 percent. of the salt with an efficiency of 60 percent. If more cells were used with magnesium cathodes, the efficiency would be increased owing to the decrease in the proportion of graphite used as cathode.

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THE DETERMINATION OF MELTING POINTS AT LOW TEMPERATURES. II.

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Further melting point determinations have been made by means of the method previously described<sup>1</sup>. The ice point was taken, and could al-

<sup>1</sup> J. Chem. Soc. 87, 1037-42 (1905).

ways be recovered sharply, by immersing the two thermo-junctions in finely chopped ice contained in a silver cylindrical Dewar vessel, 6 cm. in diameter, and 30 cm. high. The ice water collects at the bottom, leaving the ice in the upper portions, clean and just melting.

*Propyl alcohol*, *Isobutyl alcohol* and *Amyl alcohol*, have so far not been obtained in the crystalline state.

*N-Propyl Iodide*. A sample from Kahlbaum was dried 4 days over calcium chloride, and distilled. b. p. (uncor.) 101.2-101.3°. The mean of three determinations gave  $-98.8^{\circ}$  as the melting point.

*Isopropyl Iodide*. A sample from Kahlbaum, dried over calcium chloride, boiled at 89.0-89.4 (uncor.). It freezes to a more or less turbid mass, and melts between  $-89.0^{\circ}$  and  $-91.8^{\circ}$ .

*Isobutyl Iodide*. A sample from Kahlbaum was dried over calcium chloride, freed from iodine by being placed in contact with a strip of bright copper foil, and distilled, the bulb of the thermometer being wrapped round with cotton wool. It started to boil at 118-118.6°, and the main fraction between 119.3-119.6° (uncor.) was collected. Isobutyl iodide freezes glassy, and the mean of 4 determinations gave  $-90.7^{\circ}$  as its melting point.

*Isoamyl Iodide* (Kahlbaum) was not obtained in the crystalline state.

*Ethyl Formate*. A sample was distilled after drying with calcium chloride, thermometer wrapped in cotton wool. b. p. 53.5°. Three determinations gave  $-78.9^{\circ}$  as the melting point.

*Ethyl Acetate*. A pure sample (K) from Kahlbaum was dried with calcium chloride, and distilled. Three determinations gave m. p. =  $-82.8^{\circ}$ .

*Ethyl Propionate*. Dried over calcium chloride, distilled, thermometer wrapped in cotton wool (low barometer). b. p. 98.5-98.6° (uncor.). Two determinations gave m. p. =  $-72.6^{\circ}$ .

*Ethyl Butyrate*. No constant boiling preparations were at first obtainable, although specimens were obtained from various dealers. Finally, a special (K) sample procured from Kahlbaum boiled steadily at 118.9°-119.1°. This ester at first freezes to a glass, and crystallizes on warming. Two determinations gave m. p. =  $-93.3^{\circ}$ .

*Ethyl Valerate* could not be made to crystallize.

*Methyl Acetate* distilled after drying with calcium chloride, boiled at 56.5°-56.9°. The mean of three melting point determinations was  $-98.7^{\circ}$ .

*Propyl Acetate*. A sample (K) from Kahlbaum, dried with calcium chloride, distilled at 101.9-102.4 (uncor.) (high barometer). Two determinations gave m. p. =  $-92.5^{\circ}$ .

*Isobutyl Acetate* could not be obtained in the crystalline state.

*Isoamyl Valerate* could not be made to crystallize.

*Pentane.* Kahlbaum's special (K) pentane when very slowly and carefully cooled, could be obtained in the form of white crystals. The melting point was determined by suspending the melting point tube containing the frozen pentane over some liquid air residue contained in the bottom of a Dewar cylinder. Two determinations gave  $-147.5^{\circ}$  as the melting point.

*Hexane.* Synthetic (K) hexane from Kahlbaum, boiled at  $68.8^{\circ}$ – $68.9^{\circ}$ . It freezes to a white crystalline solid, and its m. p. was found to be  $93.5^{\circ}$ .

*Heptane* could only be obtained as a white, vaseline-like mass when frozen, and gave no melting point.

*Octane* prepared from octyl iodide (K) Kahlbaum. The sample started to boil at  $122^{\circ}$ , and the middle fraction between  $124$ – $125.2^{\circ}$  was used for a determination, giving m. p.  $-98.2^{\circ}$  as the mean of two experiments.

*Acetaldehyde.* (K) from Kahlbaum. Three determinations gave as mean m. p.  $-124.6^{\circ}$ .

*Acetone.* This was purified by means of the bisulphite compound, dried over calcium chloride and distilled, b. p. =  $56.4$ . It immediately crystallizes on cooling. Three determinations gave m. p. =  $-94.6^{\circ}$ .

*Methyl Ethyl Ketone.* A sample from Kahlbaum dried over fused calcium chloride, distilled at  $77.7$ – $78.0^{\circ}$  (uncor.), thermometer bulb wrapped in cotton wool. The boiling point rose to  $78.4^{\circ}$ . Two determinations gave m. p. =  $-85.9^{\circ}$ .

*O-Xylene.* A sample was boiled for 30 minutes with sodium, and distilled, b. p. =  $142.5$ – $142.6^{\circ}$  (uncor.). Three determinations gave as mean m. p.  $-27.1^{\circ}$ .

Some of the results obtained are shown in the subjoined tables :

Methyl alcohol .....	= $-97.8^{\circ}$	Methyl iodide.....	= $-64.4^{\circ}$
Ethyl alcohol .....	= $-117.3^{\circ}$	Ethyl iodide .....	= $-108.5^{\circ}$
Propyl alcohol .....	= .....	N-Propyl iodide.....	= $-98.8^{\circ}$
Iso-Butyl alcohol.....	= .....	Iso-Propyl iodide.....	= $-89$ – $91.8^{\circ}$
Iso-Amyl alcohol.....	= .....	Iso-Butyl iodide.....	= $-90.7^{\circ}$
Methyl acetate.....	= $-98.7^{\circ}$	Iso-Amyl iodide.....	= none
Ethyl acetate .....	= $-82.8^{\circ}$	Ethyl formate.....	= $-78.9^{\circ}$
Propyl acetate .....	= $-92.5^{\circ}$	Ethyl acetate .....	= $-82.8^{\circ}$
Iso-Butyl acetate.....	= .....	Ethyl propionate.....	= $-72.6^{\circ}$
Iso-Amyl acetate.....	= .....	Ethyl butyrate.....	= $-93.3^{\circ}$
Pentane .....	= $-147.5^{\circ}$	Ethyl valerate .....	= .....
Hexane.....	= $-93.5^{\circ}$	Iso-Amyl valerate.....	= .....
Heptane.....	= .....	Acetaldehyde .....	= $-124.6^{\circ}$
Octane .....	= $-98.2^{\circ}$	Acetone .....	= $-94.6^{\circ}$
Ether .....	= $-117.6^{\circ}$	Methyl ethyl ketone.....	= $-85.9^{\circ}$
Ethyl alcohol.....	= $-117.3^{\circ}$		
Ethyl bromide .....	= $-117.8^{\circ}$		

A study of the tabulated melting points again shows the value of the melting point as a criterion of purity. Of the alcohols, only methyl and ethyl alcohol give a melting point and appear to be pure substances. All the others examined only set to a vaseline-like pasty mass, and are probably mixtures of the various isomers. The derivatives, such as the esters, are obviously purer, and the alkyl iodides appear to be still more readily obtainable in the pure state. Probably pure alcohols suitable for melting point determinations, could be obtained from their iodides. The melting points of  $C_2H_5O.H$ ,  $C_2H_5O.C_2H_5$ , and  $C_2H_5.Br$  are practically identical =  $-117.6^\circ$ . From the first two identities it appears as if H and  $C_2H_5$  had the same effect on the melting point. Hence if  $C_2H_5.Br = -117.6^\circ$ ,  $HBr$  should be  $-117.6^\circ$ . The melting point as given by a different experimenter =  $-120^\circ$ , a sufficiently striking coincidence.

An experiment was made to see what amount of another substance prevented total crystallization, and it was found that pure ethyl alcohol to which 2 per cent. of isobutyl alcohol had been added, only crystallized partially, the remainder setting to a hard transparent glass in which crystalline nuclei were interspersed.

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#### NOTE.

*The Determination of Aldehydes and Furfural in Whiskey.*—In a paper on this subject in this Journal (28, 1611) by Tolman & Prescott, Tolman (p. 1628) says: "Schidrowitz does not use the distillate for this determination (*i. e.*, of the aldehydes) but clarifies with basic lead acetate, etc. . . . but this method, while it is applicable to pure whiskeys light in color, cannot be applied to whiskeys that are colored with caramel, as caramel is not removed by lead acetate"; and on page 1629 refers in a similar manner to my estimation of furfural.

It might be inferred from this that I invariably use the lead acetate clarification method, and that I am not aware of its limitations, but as a matter of fact, in the paper of mine to which Tolman refers<sup>1</sup>, I made the following remarks:

(a) "At the same time it became apparent that the statements of Carles and others . . . to the effect that cask-colored spirits may be completely decolorized by means of lead acetate . . . are not correct. They certainly lose rather more color than spirits artificially colored with caramel . . . ."

(b) "*The Coloration of the Control Solution:* For this purpose caramel was not found to be practicable, as the reagent exerts a specific influence on this coloring matter."

<sup>1</sup>The Chemistry of Whiskey I. J. Soc. Chem. Ind., June, 1902.