should receive authoritative approval as a check in the analysis of normal milk it might assist in establishing the adoption of similar methods of analysis or at least in methods that gave concordant results, and would serve to correct faulty analyses.

In case the chemist obtains results not in accord with the formula which may be chosen let him regard it as necessary to investigate the cause of this disagreement.

Such a course would prevent loss of public confidence in milk analysis which at present is likely to occur.

IRIDIN, THE GLUCOSIDE OF THE IRIS ROOT.[1]

BY G. DE LAIRE AND FERD. TIEMANN.

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IN the dried rhizomes of iris florentina we have discovered a new glucoside of peculiar composition and remarkable properties. It is prepared as follows:

The alcoholic extract from ten kilos of powdered iris root is stirred with two liters of lukewarm water and one liter of a mixture of acetone and chloroform of 0.950 specific gravity.

If allowed to stand quietly the liquid separates into two layers, a lower aqueous solution of grape sugar, organic acids, coloring substances, etc., and an upper acetone-chloroform solution containing the substances of the alcoholic extract insoluble or only slightly soluble in water.

The glucoside extracted from the root by means of the alcohol swims in the dark-colored syrup in amorphous white masses. The two layers are separated by decantation, the white flakes collected on a filter, washed with a little hot water and dried at 100°. The white powder obtained in this way was washed with ether and light petroleum to free it from adhering impurities and crystallized from boiling dilute alcohol (one volume ninety per cent. alcohol to two volumes water). We call the substance obtained in this way

Iridin ($C_{24}H_{26}O_{13}$). It forms fine white needles which become yellow in moist air and melt at 208°. It dissolves scarcely at all in alcohol, somewhat more easily in acetone. At ordinary

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[1] This paper was intended for the World's Congress of Chemists, but arrived too late for presentation.

temperatures one liter of alcohol dissolves about two grams, one liter of acetone about thirty grams. The substance is insoluble in ether, ethyl acetate, benzene, and chloroform, but dissolves easily in hot alcohol. Chloroform precipitates it from its solution in acetone, and the mixture of acetone and chloroform of 0.950 sp. gr., which we used in its preparation, dissolves only five grams per liter. At ordinary temperatures iridin is not attacked by dilute mineral acids. By the action of aqueous alkaline solutions deep yellow solutions are obtained from which, after a short time, the unchanged substance can no longer be separated by acids.

Iridin has the composition expressed by the formula, $C_{24}H_{26}O_{13}$.

ANALYSIS.

	C.	H.
Calculated percentage	55.17	4.98
Found........................	55.06, 55.23, 55.19	5.25, 5.27, 5.47

By means of dilute alcoholic sulphuric acid, iridin is decomposed at 80°–100° according to the equation

$$C_{24}H_{26}O_{13} + H_2O = C_6H_{12}O_6 + C_{18}H_{16}O_8$$

into grape sugar and a well crystallizing substance which we call irigenin.

To effect this decomposition a mixture of thirty parts glucoside, thirty-five parts water, three parts concentrated sulphuric acid, and forty-five parts alcohol is heated in pressure flasks for five to six hours on the water bath with occasional shaking. The liquid is decolorized as far as possible with animal charcoal and set aside to crystallize. The largest part of the irigenin soon separates as yellowish crystals; the rest crystallizes out after the alcohol has been driven off by a current of steam. The average yield of irigenin was sixty-five per cent., while theory requires 77.77 per cent.

The glucose is found in the liquid filtered from the irigenin, which contains also all the sulphuric acid used for the decomposition. This is removed by boiling with barium carbonate and the filtrate evaporated. The syrup so obtained solidifies after standing several weeks in crystals which are purified by recrystallization from methyl alcohol. We find the melting point of the anhydrous crystals to be 145°, of its osazone 205°. The aqueous solution of the sugar is dextro-rotatory and shows birotation. Using boiled solutions we obtained

$[\alpha]_D = 8.11°$ at 20°
$c = 7.052$, $l = 220$ mm.,
and sp. gr. = 1.0296,
therefore $[\alpha]_D = 52° \; 54'$

while according to Tollens formula $[\alpha]_D$ for a seven per cent. grape sugar solution is 52° 40′. The sugar is, therefore, *d*-glucose. We have found no other sugar in the syrup from iridin.

Irigenin ($C_{16}H_{18}O_8$).—Irigenin may be purified without difficulty by repeated crystallization from boiling benzene. Water separates it from an alcoholic solution in distinct rhombohedra. The crystals melt at 186°; alcohol, benzene, and chloroform dissolve it easily when warmed, ethyl acetate and chloroform at the ordinary temperature. It is, however, difficultly soluble in water and nearly insoluble in ether and light petroleum. It has the properties of a phenol. Even very dilute alcoholic irigenin solution is colored deep violet by ferric chloride solution. Alkalies dissolve irigenin but decompose it rapidly, which is made evident by the deepening color of the yellow solution. After a short time acids no longer precipitate from the alkaline solution an unchanged compound, but an amorphous precipitate.

ANALYSIS OF CRYSTALLIZED IRIGENIN.

	Per cent. C.	Per cent. H.
Calculated for $C_{18}H_{16}O_8$	60.00	4.44
Found	59.79, 59.78, 59.48, 59.77	4.50, 4.48, 4.54, 4.46
"	60.27	4.13

The rise in boiling point by Beckmann's method with an alcoholic solution is in accordance with this formula.[1]

Irigenin contains two hydrogens easily replaceable by acyles, which indicates the presence of two hydroxyls.

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[1] Beckmann's method for molecular weight determinations usually gives with compounds of complex composition like irigenin, using concentrated solutions, results which are too high. Under these conditions we have found the molecular weight one-fourth to one-third too high. In order to facilitate the dissociation of the molecular aggregates the solution may be diluted, with, however, a correspondingly smaller difference in boiling points. These vary too much with the use of the ordinary laboratory apparatus to allow one to obtain satisfactory figures for the molecular weight.

Dibenzoylirigenin, $C_{18}H_{14}O_8(COC_6H_5)_2$, was prepared by the Baumann-Schotten method by the action of benzoyl chloride upon a freshly prepared solution of irigenin in dilute alkali. It is necessary to work rapidly and at a low temperature so that the reaction may take place before the irigenin is decomposed in another way by the alkali. But the formation of by-products can not be entirely avoided. They are separated by repeated fractional precipitation from the benzoyl solution by means of light petroleum. The substance is finally recrystallized from alcohol and forms a white crystalline powder melting at 123°–126°.

ANALYSIS.

	Per cent. C.	Per cent. H.
Calculated	67.61	4.22
Found	67.08	4.40

Diacetylirigenin, $C_{18}H_{14}O_8(COCH_3)_2$, is easily formed by heating in a closed tube for three hours five parts irigenin with five parts melted sodium acetate and ten parts acetic anhydride. It separates in crystals when the viscous product of the reaction is diluted with water and neutralized with soda. The solution in chloroform diluted with much ether leaves on evaporation a white crystalline powder melting at 122°, which is easily taken up by hot alcohol, dissolves only slightly in ether and is insoluble in water and benzene. It deliquesces, however, immediately if a drop of chloroform is added. Light petroleum precipitates from the chloroform solution beautiful white plates which melt at 82°, and are a double compound of diacetylirigenin and chloroform. The greater part of the chloroform volatilizes when the crystals are allowed to lie exposed to the air for some time, but the melting point does not change. The analysis of the substance recrystallized from much ether and melting exactly at 122° has led to the following numbers:

CALCULATED FOR $C_{22}H_{20}O_{10}$.

	Per cent. C.	Per cent. H.
Calculated	59.46	4.50
Found	59.33	4.71

With reference to the determination of the number of replacing acetyl groups, we have saponified the acetyl derivative with dilute alkali, and titrated the acetic acid, separated after acidifying with sulphuric acid and distilling with steam.

We noticed beforehand that irigenin was decomposed by concentrated alkali with formation of formic acid. We determined, therefore, how much formic acid was produced by treatment of a quantity of irigenin equivalent to the diacetylirigenin used for the acetyl determination under exactly the same conditions and subtracted the value obtained from that given in the acetic acid titration. For our purpose the results so obtained are sufficiently near the truth, but under these difficult conditions results agreeing closely with theory are not to be expected.

ACETYL DETERMINATION.—CALCULATED FOR $C_{18}H_{14}(C_2H_3O)_2O_8$.

	Per cent. C_2H_3O.
Calculated	19.37
Found	21.41

The compound melting at 122° is, therefore, diacetylirigenin.

(To be continued.)

PATENTS OF INTEREST TO CHEMISTS.

EDITED BY ALBERT H. WELLES.

Zinc.—Johannes Pfleger has an electrolytic process for zinc (495,637). It is described as consisting in adding a basic zinc salt solution to a zinc-containing anode, to which basic zinc solution a conducting neutral salt has been added. George T. Lewis treats roasted zinc sulphide ores by adding sodium nitrate or its equivalent to unite with the sulphur left, roasting to form soluble sulphate, leaching out, oxidizing the zinc in the ores thus freed from sulphur and collecting the zinc oxide fumes (495,593). G. M. Gouyard concentrates zinc bearing sulphides by roasting the ores, and at the final stage employing a low heat, adding a small per cent. of finely pulverized coal in a reducing atmosphere, which renders the iron present magnetic and precipitates the lead on the iron, and then separating the iron with a magnetic separator (495,550). Zinc oxide is prepared by the method of Carl V. Petraeus from sulphide ores by subliming the zinc, reducing the temperature of the gases and zinc oxide driven off below a bright red heat, catching the zinc oxide fumes, and finally heating at a red heat to whiten and free from sulphur compounds (496,205). W. R. Ingalls and Francis Wyatt treat complex ores as follows: First, roast to convert into sulphates, recovering sulphur driven off as sulphuric acid; next, lixiviating with water and said sulphuric acid, removing iron if necessary, precipitating zinc as carbonate or basic carbonate, using sodium carbonate, and burning to zinc oxide,