

the extractor, E (keeping this at the boiling temperature), through the hole, H, in the condenser tube into the condenser, from where the condensed solvent flows through the side tube, S, into the bottom of the extractor. The solution of wax or fat overflows from the top of the extractor down into the flask, F. After the extraction is finished the extractor may be taken out, dried and weighed to corroborate the result obtained by weighing the flask.

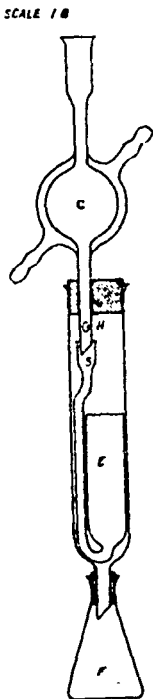
I found that I was able to do the extractions not only more quickly but also more completely with this apparatus than with an ordinary Soxhlet. It may, perhaps, also be mentioned that very little more solvent is required than is needed to fill the extractor, E, and that the bulk of it remains there when the extraction is finished.

In my case I gained yet another point by this apparatus, because I had to perform the extraction in the presence of water. This, of course, rose gradually to the surface of the solvent in the extractor and trickled down into the flask below, while in a Soxhlet it had a tendency to remain behind when the solvent emptied itself out through the syphon tube. The *modus operandi* in such cases was generally to put some water into the flask from the beginning together with the solvent. After the extraction was finished the rest of the solvent was removed by boiling the water, and on then cooling it was fairly easy to take out quantitatively the hard cake of wax floating on the surface of the water, dry it and weigh it.

It is obvious that the length of the side tube, S, should suit the purpose in view, if more than one volatile liquid be involved in the process. Supposing, for instance, that water is present and the specific gravity of the solvent is 1.5, then, if the height of the side tube be less than 1.5 times that of the extractor, the condensed water coming down from the condenser will overflow from the side tube and never enter the extractor, while if the height of the side tube be more than 1.5 times that of the extractor, the water will force its way through the solvent and overflow from the extractor.

Possibly the apparatus may be found useful for other purposes than the one mentioned. I have been able to use it with advantage for the extraction of liquids such as aqueous solutions or emulsions with solvents of lower specific gravity such as ether. It goes without saying that in such cases the side tube, S, must be high enough to allow the ether to force its way through the water into the extractor.

My apparatus have been made by Messrs. Brady and Martin, Northumberland Road, Newcastle-on-Tyne.



Given free contact with air a polluted water will absorb oxygen and eventually become purified so far as readily oxidisable matter is concerned. Therefore a determination of the amount of dissolved oxygen becomes an important factor in judging the character, say, of a stream into which effluent of a polluting character is discharged. This is borne out by the prominence which is given to dissolved oxygen in the Eighth Report of the Royal Commission on Sewage Disposal. Many methods have been suggested for determining dissolved oxygen in water but these have been designed mainly for laboratory use. A field method was proposed by Ramsay and Homfray (this J., 1901, 1072); this, however, requires special apparatus. Also the results are said to be modified by the presence of nitrites.

Mackey and Middleton published a method based on the use of pyrogallol and caustic potash (this J., 1898, 1127), and, working in Mr. Mackey's laboratory, my attention was drawn to the method with a view to adapting it to field work. It was found that the colour produced by the action of alkaline pyrogallate on the dissolved oxygen changed rather quickly and at times erratically, also that the colour produced was not exactly proportional to the quantity of oxygen present.

Pyrogallol with ammonia was tried but found useless. Various substances analogous to pyrogallol were tried in conjunction with caustic potash, i.e., gallic acid, resorcinol, catechol, and quinol (hydroquinone), but none of these was of use.

It occurred to me that advantage might be taken of the colour produced by the action of potassium thiocyanate on ferric salts. Ferrous sulphate and caustic soda were added to a water under a layer of toluol, then acidified with hydrochloric acid and potassium thiocyanate added. The colour produced was matched with a standard ferric chloride solution. Good results were obtained when the oxygen was under 5 c.c. per litre, but over that amount the colour became too deep to match accurately. Also nitrites had a disturbing effect. The method was therefore discarded.

To overcome the detrimental effect of nitrites it seemed that the most likely line to work on would be one in which the ferrous iron could react, and any excess be detected, in an alkaline solution. Potassium ferrocyanide, the usual reagent for ferrous iron, is of no use in alkaline solutions, so another indicator was sought for without success until I saw a paper by Linossier (this J., 1891, 726) in which phenosafranine is used as an indicator for ferrous iron in presence of alkaline tartrate for the determination of dissolved oxygen in water. So the method I have now to describe becomes Linossier's adapted to field conditions.

Linossier's original process is essentially for the laboratory, involving the use of 400 c.c. of mercury, the water under examination being completely isolated from contact with the air, and is designed to give accurate results. I have found though that with very little apparatus it gives sufficiently accurate results, and, as a field method, may be used so as to give a determination in a minute or two, thus enabling one to make a large number of determinations and decide as to the condition, say, of a stream at different points, and to follow up at once any clue as to pollution.

The method I adopt is as follows:—

To 50 c.c. of the water contained in a 100 c.c. Nessler cylinder are added 5 c.c. of alkaline tartrate such as is used for Fehling's solution and one drop of phenosafranine solution (1 in 2000 of water), then from a 10 c.c. graduated pipette a solution of ferrous sulphate (0.22 gm. of pure FeSO_4 and 1 c.c. of conc. sulphuric acid in 100 c.c.) is run in just below the surface of the liquid stirring gently with the pipette until the colour is discharged, looking through the cylinder horizontally. The ferrous sulphate is added 1 c.c. at a time until the colour begins to fade then in additions of 0.5 c.c. to the end. There is a slight colouration at the surface of the liquid, but if the stirring is not too vigorous this does not interfere. The outlet hole of the pipette should not be more than 1 mm. in diameter to minimise diffusion.

Yorkshire Section.

Meeting held at The University, Leeds, on Monday, January 20th, 1914.

MR. GEORGE WARD IN THE CHAIR.

A FIELD METHOD FOR DETERMINING DISSOLVED OXYGEN IN WATER.

BY JAMES MILLER, F.I.C.

As the pollution of a water by organic matter increases, the amount of oxygen held in solution decreases until a point may be reached where the latter is nil.

The pipette reading gives the cubic centimetres of oxygen per litre. Theoretically 1 c.c. ferrous sulphate of above strength does not equal 1 c.c. oxygen per litre working on 50 c.c. of sample (0.25 per cent. FeSO_4 is theoretical strength) but under the conditions stated I have found that the above strength gives correct results.

Each experimenter should standardise his ferrous sulphate against water of known oxygen content, say distilled water shaken with air until saturated, taking the temperature and referring to Roscoe and Lunt's table (Sutton's Volumetric Analysis, page 260) for the amount of dissolved oxygen present.

In the following table are given some results by this (the field) method, compared with results obtained by the Winkler method:—

	Winkler.	Field method.
	c.c.	c.c.
Leeds supply	7.8	8.0
River Aire	7.0	7.5
River Aire	4.0	5.2
Effluent	2.0	2.5

Normal, 2-normal, and 3-normal solutions of sodium chloride contain, according to Winkler (this J., 1911, 382 and 644) at 16° C., 4.7 c.c., 3.4 c.c., and 2.4 c.c. of oxygen per litre respectively. By the field method, 5.0 c.c., 3.5 c.c., and 2.5 c.c. were obtained.

The ferrous sulphate keeps well. After keeping in a bottle repeatedly opened, and nearly empty, for four weeks, only 0.67 per cent. of the total iron had become oxidised. Experiments on samples containing up to 60 parts of sodium nitrite per 100,000 showed that nitrites had no effect on the method.

As dissolved oxygen plays an important part in the extraction of gold from gold ores by cyanide solutions, the method was tried on a 0.5 per cent. solution of potassium cyanide, but no end reaction could be obtained; evidently the ferrous sulphate combines with the cyanide.

DISCUSSION.

Mr. W. McD. MACKEY wrote that he had used the method for practical work in the field, for determining the extent and persistence of pollution of a stream from a known source. He had found the manipulation was easy, and the time required for a test to be only about two minutes.

Dr. H. T. CALVERT said that he had worked the process on the River Calder, and he could bear out all that Mr. Miller said. The methods at present in use would be divided into two classes, those suitable for very clean waters, and those suitable for dirty ones. Ramsay's method was only of use for very clean waters. Winkler's method was of almost universal application. The method advocated by Mr. Miller generally gave results rather lower than Winkler's method. They had adopted Winkler's method because it was accurate and easily carried out. The apparatus only weighed five or six pounds, and in order to simplify the operations in the field, they could proceed as far as the precipitation of the manganese hydroxide, and then finish the estimation in the laboratory.

Mr. WARD asked how the colour of the water would affect the results.

Mr. MILLER replied that a pale colour of the water had no effect whatever on the results. Any water could be tested so long as it was not pink. He had determined the dissolved oxygen in an effluent which had a distinct pale blue colour, and the end point was quite sharp.

Journal and Patent Literature.

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German.—1 mark each (with full particulars) to Kaiserlich Patentamt, Berlin, Germany.

I.—GENERAL PLANT; MACHINERY.

PATENTS.

Extraction of liquids or solids held in suspension or combined in gases or fumes. J. Armstrong, London. Eng. Pat. 569, Jan. 8, 1913. Addition to Eng. Pat. 821, Jan. 10, 1912 (this J., 1913, 184).

THE apparatus described in the principal patent is improved in that the compressed gases are passed in succession through a number of nozzles immersed in different liquids, each adapted to separate out a particular impurity; the nozzles are carried on frameworks fixed to detachable lids, so that they can be removed for cleaning; the chambers may be of cylindrical form with radial or tangential nozzles for the gas and conical collecting chambers for the impurities beneath.—H. H.

Distillation of liquids; Process for the —. Naamlooze Vennootschap Maatschappij Destillator. Fr. Pat. 461,822, June 25, 1913. Under Int. Conv., June 26, 1912.

HEATED water from engine or other condensers is fed into an evaporator in a finely divided condition and is exposed to a vacuum so that part of the water is evaporated by its own heat, the vapour passing to a condenser also under vacuum.—W. H. C.

Measuring the flow of fluid through a conduit; Instruments for —. The British Thomson-Houston Co., Ltd., London. From General Electric Co., Schenectady, New York, U.S.A. Eng. Pat. 2417, Jan. 29, 1913.

THE pressure difference in a Venturi tube, Pitot tube, or similar measuring device is indicated by means of a mercury gauge supporting a short column of an "opaque substance such as carbon tetrachloride" contained in a narrow glass tube. The upper end of this tube is connected with the low-pressure side of the Venturi tube, while the lower end, which is enlarged, dips into a vessel of mercury connected with the high-pressure side. A permanent record of the pressure differences is made by a marking point carried by a lever attached to a U-shaped magnet, which encloses the vessel of mercury and moves in conjunction with a magnetic body floating on the surface thereof.—H. H.

Distilling feed water for boilers; Process and apparatus for —. Soc. d'Exploitation des Procédés Évaporatoires, Système Prache et Bouillon, Paris. Eng. Pat. 1583, Jan. 20, 1913. Under Int. Conv., Jan. 19, 1912.

STEAM from the boiler, 1, enters the injector, 10, by the pipe, 11, and withdraws steam from the evaporator, 6, compresses it and injects it into the heating arrangement, 7, of the evaporator; the steam is condensed and the condensed water flows into the tank, 3. The impure water