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THE MANUFACTURE OF LIGUID OXYGEN AND ITS

USE AS AN EXPLOSIVE,

F. K. MIDDLETON HUNTER, ('23).

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THESIS

Submitted to the Faculty of the

SCHOOL OF MINES AND METALLURGY OF THE UNIVERSITY OF MISSOURI,

At Rolla, Missouri,

In fulfilment of the work required for the

Degree of

ENGINEER OF MINES.

1927,

by

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By

F. K. Middleton Hunter, '23.

CHARACTERISTICS:

Liquid oxygen is chiefly characterized by its extremely low temperature, -183°C. It has the appearance of water, and when unconfined in ordinary temperatures it boils vigorously, giving off quantities of oxygen gas and behaving exactly as does water at its boiling point. At the boiling point, i.e., -183°C, its specific gravity is 1.14, or slightly heavier than water. <u>Per se</u> liquid oxygen is not explosive, needing the addition of carbon in some form before it can even burn.

HISTORICAL:

Oxygen was first liquefied in 1877, but the first record of its use commercially as an explosive was in 1899 when part of the famous **B**implon tunnel between Italy and Switzerland was driven with this explosive. Owing to the difficulties of storing and using this liquid, little more progress was made until the Great War, when Germany, forced to experiment with any promising explosive because of her rapidly diminishing stocks of Chilean nitrates, made considerable headway in the technique of using liquid oxygen.



Previously, in 1904, Sir James Dewar in England perfected a double walled vessel with a high degree of vacuum between the walls, and this was at once proved useful as a temporary storage container for liquid oxygen.

Since the War liquid oxygen explosives have been used extensively in France and Germany and to a lesser extent in England, Scandinavia, Italy and the U.S. It has also been, or is being, used in at least one mining property each in Mexico (Fachuca) and Chile (Chuquicamata), and was experimented with at Cerro de Pasco, Feru.

MANUFACTURE:

The two most common types of machines for producing liquid oxygen at present are the Linde (German) and the Claude (French). Of these, the Claude is generally supposed to be the most efficient.

Principles:

Atmospheric air is composed of approximately 21% oxygen, 78% nitrogen, and 1% inert gases, mainly argon. At atmospheric pressure, air will liquefy at -193°C, but by raising the pressure to 33 atmospheres, it will liquefy at -143°C. Briefly it may be stated that air may be liquefied within the following limits:

> Pressure, 33 atmospheres or more at -143°C. Pressure, less than 33 atmospheres at a colder temp-

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erature than -143°C.,

or: the lower the pressure, the greater the cooling necessary to achieve liquefaction.

It is well known that when air (or any gas) is compressed, it generates heat; when expanded again, it becomes colder. Also that when a gas is expanded against resistance so as to perform work (as in the cylinder of an expansion engine) a still greater degree of cooling is attained. These are the principles of the Claude system, which we propose to describe. In the Linde system the principle of the cooling effect of expanding a highly compressed gas through a nozzle or expansion valve is used; in this system much higher pressures are needed.

Practice:

In general the Claude 75 litre per hour apparatus, which is the one we shall describe, consists of:

1 Three-stage air compressor, complete with intercooler and aftercooler.

2 Oil separators.

1 Caustic soda tower, tank, and pump.

1 Intermediate pressure dessication battery.

1 High pressure dessication battery.

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1 Set of two heat exchangers.

1 Expansion engine.

1 Surpressor, or Recompressor.

1 Liquefaction column.

1 Oxygen, or rectification column.
The operation is as follows:

Soda Tower:

1. The compressed air from the three-stage compressor at 853 lbs. goes through an oil separator, and from there to the decarbonating soda tower which it enters near the bottom. Just before reaching the soda tower, the diameter of the pipe is decreased considerably so as to increase the velocity of the air. The tower is filled with annular rings (Rashig Rings) and over and through these a stream of caustic soda solution is constantly dripping. The air is forced up through these rings and by coming in contact with the caustic soda solution it is washed free (or almost free) of any carbon dioxide, dust and remaining oil particles which may be present in the air.

I.P. Dessication Battery:

2. From the soda tower, the air, still at 853 lbs., enters the intermediate pressure dessication battery. This

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is a series of two drain bottles, which have nothing in them, and twelve other bottles, in two alternate batteries of six each, full of caustic soda on perforated trays. The two drain bottles (sometimes called purge bottles) are the first and last respectively. The air, entering the first drain bottle loses its velocity and tends to drop any particles of water or caustic soda which may have been picked up in the By means of a three-way valve the air is then soda tower. sent through one of the six-bottle batteries, where any remaining traces of moisture are absorbed. The reason for having two alternate batteries of six bottles each is, of course, to assure uninterrupted operation when it becomes necessary to renew the supply of sodium hydroxide. After leaving the six-bottle battery the air goes through the second drain bottle where it drops any particles of soda picked up in the previous bottles. The two drain bottles are purged every few hours through needle valves in the bottoms.

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The decarbonating and dessicating batteries are indispensible, as otherwise the air would have carbonic acid gas and moisture in it which would form carbonic acid ice and water ice at the low temperatures which are reached, and this would clog the pipes and eventually bring the whole apparatus to a stop.

3. Leaving the intermediate pressure dessicators the main air current splits into two parts, which we shall call the A and B currents (See schematic drawing - Figure 1.)

Heat Exchangers:

A-a. The larger current (A) enters one of the heat exchangers at the top and goes down through a cluster of small copper tubes, while outside the tubes a current of very cold nitrogen, coming from the oxygen column as we shall see later, ascends. The heat exchange effected is considerable.

There are two heat exchangers, only one of which is used at a time. The air, even after the drying it has had, still contains traces of moisture, and eventually the fine copper tubes are clogged with ice and it is necessary to change the air current into the other exchanger, while the frozen one is de-rimed. Even so it is necessary to de-rime the complete set of exchangers periodically and a de-riming stove is kept for this purpose.

Expansion Engine:

A-b. After passing down through the heat exchanger, the A current goes directly to the high pressure side of the

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expansion engine where it expands from 853 lbs. to 57 lbs., and is colled in so doing. The expansion engine is coupled direct to the surpressor, and hence the air expanding in the expansion engine supplies the motive force for the surpressor and consequently is "expanding with the production of external work", and so generates a considerable degree of cold.

Coming from the expansion engine, the A current of air again splits into what we shall call the C and D currents.

C-a. The C current flows into the bottom of the middle compartment of the liquefying column (which will be discussed later) and out of the top of this same compartment. In going through it helps to cool the B current of high pressure air which, as will be seen later, travels through the liquefying column in a cluster of fine copper tubes.

C-b. On leaving the liquefying column, the C current goes into the low pressure side of the expansion engine where it expands from 57 lbs. to 2.8 lbs. generating more cold.

C-c. The C current then joins the flow of nitrogen from the oxygen column and goes to the heat exchangers where the cold of this air and nitrogen mixture is used to cool the A current of air. After passing up through the heat ex-

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changer, this air current is exhausted into the atmosphere.

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D. The D current flows directly into the lowest compartment of the oxygen or rectification column, where it is soon joined by the B current.

Surpressor:

B-a. The B current, splitting from the initial air current just before the A current goes into the heat exchangers, goes directly to the surpressor (or recompressor), which is coupled directly to, and is driven by, the expansion engine. Here the B current is further compressed from 853 lbs. to 2845 lbs.

H.P. Dessication Battery:

B-b. The B current then passes through a small oil separator and so to the high pressure dessication battery. This is composed of only two bottles, but they are fitted with trays of NaOH and are similar to the other bottles in the intermediate pressure dessication battery.

Licuefying Column:

B-c. From here the air goes to the top of the liquefying column and passes down through a cluster of fine copper tubes, around the outsides of which a counter current (upwards) of cold nitrogen (from the oxygen column) and cold air (from the high pressure side of the expansion engine - A current) is flowing.

B-d. The cooling effect is such that this air (B) which is at 2845 lbs. liquefies at a temperature close to the critical (i.e., -143°C), and this liquid flows out at the bottom and into the lower compartment of the oxygen column, where it falls over a series of platforms to a sump at the bottom.

Oxygen Column:

Here the liquid gives off vapours which are richer in nitrogen than air, for nitrogen is more volatile than oxygen and consequently evaporates first.

4. In the meantime, as shown above, the D current of air has entered the oxygen column at the bottom, and mixes with the vapours given off by the liquid air. This mixture, being at a pressure of 57 lbs., is much warmer than the liquid air and rises through a cluster of fine copper tubes. These tubes are immersed in liquid oxygen at a pressure of 4.3 lbs. and consequently at a temperature of about -130°C.

5. In the tubes there now takes place a rectification.

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There is an exchange of heat between the rising vapours and the surrounding liquid oxygen, which vapourizes some of the liquid oxygen outside, and condenses some of the oxygen in the rising vapours. This condensed oxygen falls and in so doing helps to rectify some more of the ascending vapours, and finally falls into the sump at the bottom. This liquid in the sump gradually becomes richer and is commonly called the "Rich Liquid".

6. The vapours which succeed in passing through the tubes are now collected in a hood and pass into a condensing coil which is immersed in a liquid colder than that which surrounded the cluster of fine copper tubes, for it contains a greater amount of nitrogen. These vapours, containing almost pure nitrogen, now condense and form what is known as the "Poor Liquid", because it contains only a very small percentage of oxygen.

7. These two liquids, the "Rich" and the "Poor", being at a pressure of 57 lbs., while the pressure in the upper part of the column is only at 3.6 lbs., they will rise automatically, and do so, entering near the top of the column and dripping down over platforms in such a way as to

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come into intimate contact with the ascending vapours from the bath of liquid oxygen surrounding the vapourizing tubes. There follows a complete rectification since these vapours reach a temperature at which the oxygen will temain as a liquid, while the nitrogen will vapourize. The liquid oxygen collects in the vessel through which the vapourizer tubes pass, near the bottom of the column, and is drawn off through a valve.

8. The system not being cold enough to keep the nitrogen in a liquid state, it continues to rise and escapes from the top of the column whence it returns to the liquefying column. Here some of it goes completely through the column and exhausts into the atmosphere at the top, while some only goes through the bottom compartment and thence to the heat exchangers, as shown on the accompanying schematic drawing.

STORAGE:

When it is remembered that the boiling point of liquid oxygen is nearly 300°F. below zero, it can be realized that 'ordinary room temperatures are, in comparison, extremely hot. When too it is understood that by pressure alone it is im-

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possible to prevent liquid oxygen evaporating, some idea of the difficulties in storing such a liquid may be imagined. It was mentioned above that in 1904 Sir James Dewar invented a vacuum flask, and up to the present day the Dewar flask, or one of several modifications, is the standard and most practicable method of storing liquid oxygen.

The commonest form of container is a sperical vacuum bottle of metal, having a long neck of small diameter. This is, of course, in an outside covering or box fitted with handles for easy manipulation. The commonest sizes are from 5 to 50 litres. In the 50 litre size a loss by evaporation of 2.5% of the total contents, in twenty-four hours, may be expected, while for the 5 litre size, as much as 10% will be lost. Large vacuum storage tanks are now being tried at a large South American property, which are made of steel, hold 1000 gallons each, and are expected to cut the evaporation loss eventually to 2%, including filling.

CARTRIDGE MATERIALS:

As stated above, liquid oxygen per se is not explosive, and a great deal of time and money has been spent determining the most suitable materials out of which to make cartridges. The composition of cartridges has run the gamut of oxidizable materials. At present U.S. practice seems to favour a lamp black and carbon mixture, or a straight carbon, while in France and England the tendency seems to be to include some (secret) proportions of various powdered metals or chemicals, apparently with the idea of raising the heat of oxidation and hence the rate of detonation.

The chief desideratum is to get a substance or mixture which will absorb the greatest possible amount of liquid oxygen, hold it for the greatest possible length of time, and be as nearly as possible completely oxidizable itself.

A cartridge of fine lamp black absorbs a large amount of liquid oxygen and holds it well, but the cartridge tends to be flabby and easily broken when loading. A cartridge of straight carbon, on the other hand, absorbs about as much liquid oxygen, but loses it very rapidly when removed from the soaking container, as the large interstices allow capillarity full sway, which is not the case in a cartridge of lamp black of almost colloidal fineness. But the carbon makes a nice stiff cartridge, easy to handle and load. A mixture of 65% coarse carbon and 35% fine lamp black has

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been used and has proved very successful.

CARTRIDGE COVERING:

In France a stiff paper is commonly used for making cartridges; this is used to some extent (mostly experimentally in the U.S. But a covering or case of canvas is perhaps most practical. Canvas is bought in the form of hose, of the required diameter. It is then cut into standard lengths and one end of each length clipped. The cartridge is then turned inside out, filled with the cartridge material, and clipped at the other end. With one cartridge packing machine and one clipping machine, one man and his helper can easily turn out 300 7"x20" cartridges in an eight hour shift.

SOAKING BOXES:

The chief requirement in a soaking box is insulation against heat without excessive bulk. This is not difficult of achievement if it is proposed to keep the soaking boxes on a flat car or other form of conveyance. In the United States lately a tropical wood known as Balsa has begun to come into prominence as an insulating material suitable for refrigerating cars and boxes. It is a **wood** of extreme porosity, 92% being air spaces. The air spaces are spherical in shape and not connected.

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A soaking box should be large enough to accomodate a convenient number of cartridges. If a large cartridge is in use, such as 7"x20", a box should hold 50. A box six feet long and three feet six inches wide, internal dimensions, will take 50 7"x20" cartridges standing on end in ten rows of five each. Such a box should be made of 4" balsa wood, coated on each side with a hot paraffin waterproofing coat. The inside is then lined with thin copper sheeting, and the outside covered with a protective box of 1-1/2" hard-This in turn should be waterproofed and then painted wood. white to cut down heat by radiation. The lid should be similarly constructed.

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SOAKING:

With a 7"x20" cartridge of 65% carbon and 35% lamp black, in a canvas bag, one may consider the whole as being carbon. Such a cartridge, depending on the kind of carbon, etc., will weigh in the neighbourhood of 8 lbs., and theoretically needs to soak up an additional 8x2.67 lbs. of liquid oxygen to oxidize all the carbon to CO_2 , or 8x1.33 lbs. to oxidize all the carbon to CO. In practice we consider these ratios of oxygen to carbon as 2.5 and 1.25, because our carbon contains some impurities and also the canvas is not strictly speaking carbon, so that some of our gross weight (in our case 3 lbs.) is not carbon.

Experimentally we have found that the soaking ratio of the particular carbon cartridge which we are considering, having a density of 0.34, is 3.5. In other words it will soak up 8x3.5 lbs. oxygen = 28, and will then weigh 28 plus 8, or 36 lbs.

Cartridges are soaked by placing them in an ordinary soaking box, as described above, on end, and pouring in liquid oxygen. The liquid should be poured in very slowly at first, to give the box and cartridges a chance to cool down, as otherwise startlingly Marge amounts of liquid oxygen will be lost by evaporation. It is estimated that even when filling soaking boxes slowly and with care, as much as 30% of the liquid poured in is lost in this manner.

If it is attempted to soak the cartridges to their theoretical limit a large amount of liquid will be left over in the boxes after the soaking operation is over, and this liquid is difficult to save. Consequently a compromise is effected by experimentally finding out the economic limit of

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soaking: if the cartridges are soaked fully, too much oxygen is lost; if the cartridges are not soaked sufficiently, they will not have the requisite explosive strength. Ordinarily, if liquid is continuously poured into the boxes until the cartridges weigh 28.5 lbs. and there is about six inches of liquid in the boxes, good results are obtained, and there will be found only about an inch or an inch and a half in the boxes when all the cartridges are removed. At 28.5 lbs. the cartridges will have soaked up 2.56 lbs. oxygen per lb. carbon, and there will be some forty minutes leeway to shoot in before the explosive strength will have diminished too much.

EVAPORATION CURVE:

We have also found experimentally (See Figure 2) that this same cartridge will reach the CO_2 point (i.e., the point where there is only just enough oxygen to permit complete oxidation to CO_2) in forty-two minutes.

The graph shown in Figure 2 and the remarks in these few paragraphs apply only, of course, to the particular cartridge under discussion. The diameter and size of a cartridge directly affect the evaporation rate.

It should be noted here that it is important in L.O. X. (Liquid Oxygen Explosives) blasts underground to fire the blast before the CO₂ point is reached so as to avoid the danger of CO gas and its often fatal consequences; but in open cut work, which is the condition under which we have studied the use and possibilities of L.O.X., the CO₂ point is not particularly important, as CO gas is rapidly dissipated in open air. The important thing in open cut work is ti fire the blast before the explosive strength has materially diminished.

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A glance at Figure 2 will show that wereas the evaporation is most rapid for the first hour and a half, the explosive strength diminishes very gradually at first, and more rapidly later, and that though the CO_2 point is reached in forty-two minutes, the explosive strength is not materially decreased for an hour or a little more.

It should be noted here that the evaporation rates shown were obtained in a borehole (i.e., confined) and that in the open air the rate would be considerably quicker.

While on the subject of evaporation, the following percentage losses may be of interest: Loss due to evaporation,

These values are, of course, plus or minus, but in the aggregate pretty close to results obtained. In other words only about 40% of the liquid manufactured is actually used for useful work (i.e., breaking ground.)

BLASTING:

Calculations:

First the ground to be blasted must be rated at so many tons per lb. of L.O.X. at the time of firing. This can only be down after considerable experience, but we have found that if ground is rated one tenth heavier per lb. of L.O.X. than for dynamite, good results are obtained. Having decided at what "factor" (i.e., tons per lb. L.O.X. at time of firing) to use, it is a simple matter, using the experience how long it will be after cartridges are removed from soaking boxes until the moment of firing the blast, to calculate how many cartridges will be required per hole.

As an example, take a blast having the following data:

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Class of ground . hard. Height of face . . • • • • • • 36 ft. Number 8" churn drill holes 35 Average depth . . 38 ft. Distance between rows 12 ft. Distance from average face 15 ft. Ground rated at 2.93 short tons per 1b. L.O.X. at time of firing.

Now from our graph (Figure 2) we find that the 2.56 ratio is opposite the 36 minute mark, and if we add 30 minutes to this, we have one hour and six minutes, and know that at the time of firing the blast, we shall have a ratio (1). The writer has seen L.O.X. blasts up to 33,000 s. tons.

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of 2.15, and that our cartridges will weigh (2.15x8) plus 8, or 25.2 lbs. And so it is onvious that the number of L.O.X. cartridges to load per hole (i.e., per 465 s. tons of burden) is:

1 cartridge weighs . . . 25.2 lbs.
For 1 lb. we can break . . 2.93 tons.
And for 25.2 lbs. we can break 2.93 x 25.2 tons.
Burden per hole is 465 tons.

So
$$\frac{465}{2.93 \times 25.2}$$
 = 6.3 cartridges.

and in loading, two holes will be given 6 cartridges and the third, 7, thereby arriving at a correct total - for it is not practical to break cartridges in half, though it can be done.

Comparison vs. Dynamite:

It has been found by experience, blasting L.O.X. in comparison with a 67% ammonia free-running dynamite, that in hard ground L.O.X. has a greater shattering effect and less propulsive force. This is often and advantage, and is to be expected, when one considers the velocities of detonation of these two particular explosives:

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The dynamite in question . . . 3700 metres per sec. The particular L.O.X. mixture . . 5200 " " "., and in general it may be stated that nin hard ground, the higher the rate of detonation the better the breakage, and the slower the rate of detonation, the greater the throw.

Methods of loading and Firing:

In L.O.X. blasts witnessed by the writer in France ⁽²⁾ in September, 1926, paper covered cartridges were used, and each cartridge, when brought to the blast in the soaking box, had a long string attached to the upper end (the "ear"), and was carefully lowered into the hole. Countered cordeau Bickford was laced through the second cartridge from the bottom, and the holes were spliced to the main uncountered cordeau leads in the normal manner. The blast was fired with two caps, one at each end of the main lead, by a hand blasting machine.

In some L.O.X. blasts witnessed by the writer in Indiana, in October, 1926, the cartridges were canvas covered and were merely dropped into the holes, a length of cordeau having previously been placed in each hole. At this property a separate electric blasting cap was used to detonate

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^{(2).} At the mines of Les Petits Fils de Francois de Wendel et Cie., Hayange, Lorraine.

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the cordeau in each hole, the caps all being fired simultaneously by a hand blasting mchine. This practice of using one cap per hole with cordeau Bickford is ridiculous, and not only raises costs, but actually introduces additional risks of misfires.

In some blasts in the United States, canvas covered cartridges are fitted with a hook in one end and an eye in the other, and are loaded by hooking a string of cartridges together and loading them by hand. This is an unnecessary refibement, except in very badly drilled holes, or in exceptionally fissure-y ground where cartridges may be expected to hang up or break repeatedly. In ordinary ground, this procedure only wastes time and increases the elapsed time between soaking and detonating.

At a large South American property, the cartridges are canvas covered and are dropped into the holes, which have cordeau previously placed in them. No difficulties are experienced, though cartridges are bound to hang up and break once in a while. Detonation is by the usual cordeau practice of having one cap at each end of the main leads to set up converging detonating waves.

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COSTS:

No attempt will be made to give any costs on L.O.X. at present. The largest item would be cartridge materials; then interest and amortization on the cost of the plant, which might easily amount to as much as three cents per lh. of liquid produced, depending on the number of years in which to amortize the capital outlay. The next largest item would be power, which varies greatly in cost per KWH in different parts of the world, followed by labour, superintendence, repairs and packing, chemicals, lubricants, lights, and miscellaneous, in order of importance.

TESTING:

All the usual tests applied to dynamites have been tried with greater or lesser success on L.O.X. The following notes may be of interest:

Trauzl Test:

This is used as a measure of strength, and is the net enlargement of an axial hole of known volume in a lead block of 20 cm. diameter and 20 cm. height, made by a ten gram charge. In using such a small amount of L.O.X. the difficulties encountered due to rapid evaporation are insuperable,

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and this test is not considered as of any value for L.O.X. The writer has personally spent considerable time trying to evolve a method of using this test on L.O.X., but has not been successful.

Ballistic Pendulum:

This is a canon or mortar mounted in such a way as to swing in an arc when a charge is fired in it. This test is commonly used as a measure of propulsive force. Difficulties are encountered here too, due to evaporation, but the charge used may be of considerable size, and consequently good results may be obtained. Straight carbon cartridges give the highest results and indicate a strength equivalent to 40% N.G.dynamite. In field tests, as mentioned above, the writer considers L.C.X. as superior in breaking power (the ultimate desideratum) to 40% dynamite, and, as shown here, inferior in throw.

Bichel Gauge:

This is a closed bomb, and in it explosives are exploded. By this means the volume and the pressure of the gases generated may be determined. In this test L.C.X. does not quite equal results obtained with 40% dynamite.

Velocity of Detonation:

This is determined in the usual Dautriche method using cordeau Bickford of known velocity, and a lead plate. Straight carbon cartridges give rates varying with their density, up to about 5200 metres per second.

Miscellaneous Tests:

Various other tests have been tried with greatly varying results, depending on the composition of the cartridge and its density. Such tests are:

Sensitiveness to impact. Sensitiveness to friction. Detonation by influence. Continuity of detonation. Flame test.

Inflamability.

Small lead block test.

Products of combustion.

Gallery test.

Temperature calculations.

Effect of moisture.

But the commonest tests on explosives are the four des-

cribed above, and, as shown, only three of them are applicable to L.O.X.

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CONCLUSION:

L. L.C.X., still a baby in the explosives world, is rapidly coming to the fore and will undoubtedly, before many more years have passed, occupy an important place among explosives for cutdoors use. It has been tried, but without much success, underground, and it may be that difficulties due to rapid evaporation in small diameter cartridges and the inherent danger of **CB** production may prove an effective barrier to its use for this purpose.

During a recent visit to Europe the writer visited a large steel and cement district in Moselle, France, and found, in this particular district, no other explosive being used. L.O.X. was used in open quarries with large diameter churn drill holes and with small diameter horizontal Leyner holes, besides being used for blockholing and tree stump remaval, and exclusively underground. In this district the Weber cartridge (containing chemicals) was used, which is made in different grades so as to obtain blasting results equal in effect to anything desired from black powder up to the highest N.G. dynamites. It has been said that variety is the spice of life, and work with LoO.X. has so many variables and so few constants that it makes a most fascinating field of study.

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CONVERSION FACTORS.

l Kg. per sq. cm.	equals	14.22421 lbs. per sq. in.
l lb. per sq. in.	18	0.07031 Kg. per Sq. cm.
l litre	11	0.2642 U.S.Gallons.
1 U.S.Gallon	19	3.785 litres.
1 metre per second	•	3.2308 ft. per sec.
l foot per second	11	0.3048 metres per sec.
1 short ton	*	0.8929 long tons.
l long ton	11	1.1200 short tons.
Specific gravity of	f liquid oxygen at b	ooiling point (i.e.,
-183°C) is 1.14.		

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