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October 2, 1944

CONTRACT NO. W-7405 eng. 43
TECHNICAL REPORT FOR THE PERIOD
August 1, 1944 to September 30, 1944

THE HARSHAW CHEMICAL COMPANY

UNCLASSIFIED

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Chief, Declassification Branch

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CONTRACT NO. W-7405 eng. 43
TECHNICAL REPORT FOR THE PERIOD
August 1, 1944 to September 30, 1944

THE HARSHAW CHEMICAL COMPANY

I. DEVELOPMENT AND PLANT USE OF CONTROL ANALYTICAL METHODS

A. Determination of Molybdenum

Tests were made on composite samples of the first 23 lots of C-616, using the method described in the "Technical Report for the Period May 1, 1944 to July 31, 1944, CONTRACT NO. W-7405 eng. 43", dated August 5, 1944.

Because the platinum weighing tube tended to become distorted due to pressure, a slightly longer nickel tube with heavier walls was used for many of the tests and found to be satisfactory.

For the first 23 lot samples, molybdenum was found to be present in quantities ranging from about 0.1 p.p.m. to 3 p.p.m. with all except 3 samples analyzing 1 p.p.m. or less. Because of this consistently small amount of molybdenum found, further spot tests have been made only on one lot per week, and in each case less than 1 p.p.m. was found.

B. Determination of Iron

Tests for iron in plant composite samples were carried out using either the platinum or nickel sample tube as described above. In early tests, when iron sample cylinders were used, variable quantities of iron (0 - 29 p.p.m.) were attributed to attack of the iron surface, possibly a superficial oxide film. When nickel sample cylinders came into use, however, it was presumed that the iron content of the samples would be consistently low since previous experience had proved that iron compounds deliberately added to C-616 were non-volatile and would not be expected to carry through the manufacturing and purifying operations.

Nevertheless, analyses continued to show iron in quantities varying over the same range as had been previously found. Careful blowing out of the apparatus to remove residue did not alter the results. The possibility of scale or surface attack on a freshly cleaned cylinder was considered. A cylinder was filled twice with C-616 and discharged in the inverted (valve down) position, and then refilled and tested. A liquid sample showed 9 p.p.m. iron. Since iron compounds had appeared to be non-volatile, samples were taken by slowly distilling C-616 gas from the upright cylinder and condensing the material in the sample tube. On three such tests, using different cylinders, iron was found corresponding to 12, 13, and 9 p.p.m. Careful check of reagents, sample tubes, method, etc. has yet revealed no source of error.

C. Determination of HF

Previous reports from this laboratory have described the use of three methods for determining HF in C-616. The following discussion will describe further tests by these methods and their correlation:

1. Reflux Gas Volume Method

This method, described in detail in the August 5 technical report, has been used successfully for plant testing of some forty lots of C-616.

As previously reported, this analytical procedure, if desired, can be used to differentiate between "Total Gas" and "Actual HF", the term "Total Gas" being used to include, in addition to HF, gases such as nitrogen which are non-condensible at liquid nitrogen temperatures. This ability to distinguish between the impurities has proved of considerable value, as will be described later.

Using this reflux method of analysis, plant samples in general were found to contain less than .01% Total Gas as HF. In the few cases where total gas exceeded .01%, only one was found to contain HF in quantity greater than .01% (actually .014%). In one sample Total Gas was found to more than 0.05% whereas the Actual HF was only .009%. Some further consideration will be given to this method later where it will be compared with other methods.

2. Original Freezing Point Method

Fairly consistent results have been secured using the freezing point cells and furnace described by Priest in Col. Serial 3B-L-114. Although in general, satisfactory check results were secured for a particular charge in the cell, the values obtained did not check satisfactorily those secured by other laboratories using the same or similar cells. Further early tests made by the freezing point and reflux method did not check at all well. Comparative results on standard samples supplied by Columbia have been previously reported. On plant composite samples, analyses by these two methods differed as indicated by the following results:

Sample No.	Total Gas as HF, %	Actual HF, %	HF by F.P.Cell, %,Kellex Data	
1	0.013	0.004	0.018	0.021
2	0.015	0.014	0.030	0.033
3	0.003	-----	0.013	0.015
4	0.003	-----	0.014	0.016

In each instance it will be noted that the freezing point method gives values which are consistently higher than those for even the Total Gas. These samples were taken from iron sample cylinders but the amount of iron present (not over 20 p.p.m.) would appear to be too small to affect the freezing point to any great extent.

S███████████

3. Modified Freezing Point Method

A considerable amount of data relating to this method has been accumulated since preparation of the Progress Report (Contract W-7405 eng. 43) dated August 18, 1944, describing this new technique which uses a special sample cylinder (see attached Sketch No. 092044). Basic principles of the method remain unchanged but a few modifications tend to simplify the analysis.

For the screw connections (nipple-to-cylinder and valve-to-tee) a heavy paste of litharge and Vistanex in carbon tetrachloride has proved much more satisfactory than when the litharge is omitted.

The steam line into the thermometer well has been found unnecessary during the filling operation when steam-traced lines and a steam hood over the valve assembly are used.

The sample cylinder containing material to be tested is placed in the bath at 80° to 90°C for one hour. (No tests have been made to determine the minimum time required to completely melt the C-616, but one hour has proved adequate.) After the melting period, the cylinder is transferred to the cooler bath (63.2 ± .2°C.) and allowed to remain undisturbed for 15 minutes. At the end of the 15-minute period, the cylinder is shaken vigorously in the bath by means of a loop of wire around the valve. (If necessary to induce crystallization, the cylinder may be removed from the bath and shaken although this procedure usually gives less satisfactory freezing point curves.) Twenty to 30 seconds after the first shaking the cylinder is again agitated. With this method, using the double shaking technique, recovery of temperature to the flat point is generally more rapid and a better curve is secured than when a single shaking is used, possibly due to better distribution of seed crystals.

Following the second shaking, the resistance thermometer or thermocouple, warmed to bath temperature, is inserted into the thermometer well. The temperature rise is noted visually but readings need not be taken until the temperature rise becomes slow (0.02°C per minute) and the anticipated freezing point is approached. If the previous shaking has not induced crystallization (as is infrequently the case) or if the temperature rise is slow at points considerably below the expected freezing point, the thermoelement should be removed and the cylinder again shaken.

a. Effect of Slow Crystallization

If excessively slow crystallization occurs, a very short flat portion in the curve, or none at all, may be secured in which case the test must obviously be repeated. Occasionally, however, when slow crystallization occurs - a flat portion of adequate duration may be secured. This freezing point, however, will tend to be lower than one secured using the same sample where rapid crystallization occurred.

BRASS OR COPPER PIPE
2" LONG — THR. 1 END

SILVER SOLDER

KEROTEST
VALVE #7983

SILVER SOLDER

$\frac{3}{8}$" BRASS TEE

SILVER SOLDER

$\frac{3}{8}$" x 2" BRASS NIPPLE

LITHARGE VISTANEX

NICKEL CYLINDER
APP CAPACITY - 765 CC
1.69" WATER

COPPER TUBE - 11" LONG APP.
.375 O.D. - .311 I.D.

$7\frac{5}{8}$"

$\frac{1}{2}$"

$3\frac{5}{8}$"

$\frac{1}{8}$" THICK COPPER PLUG
SILVER SOLDERED.
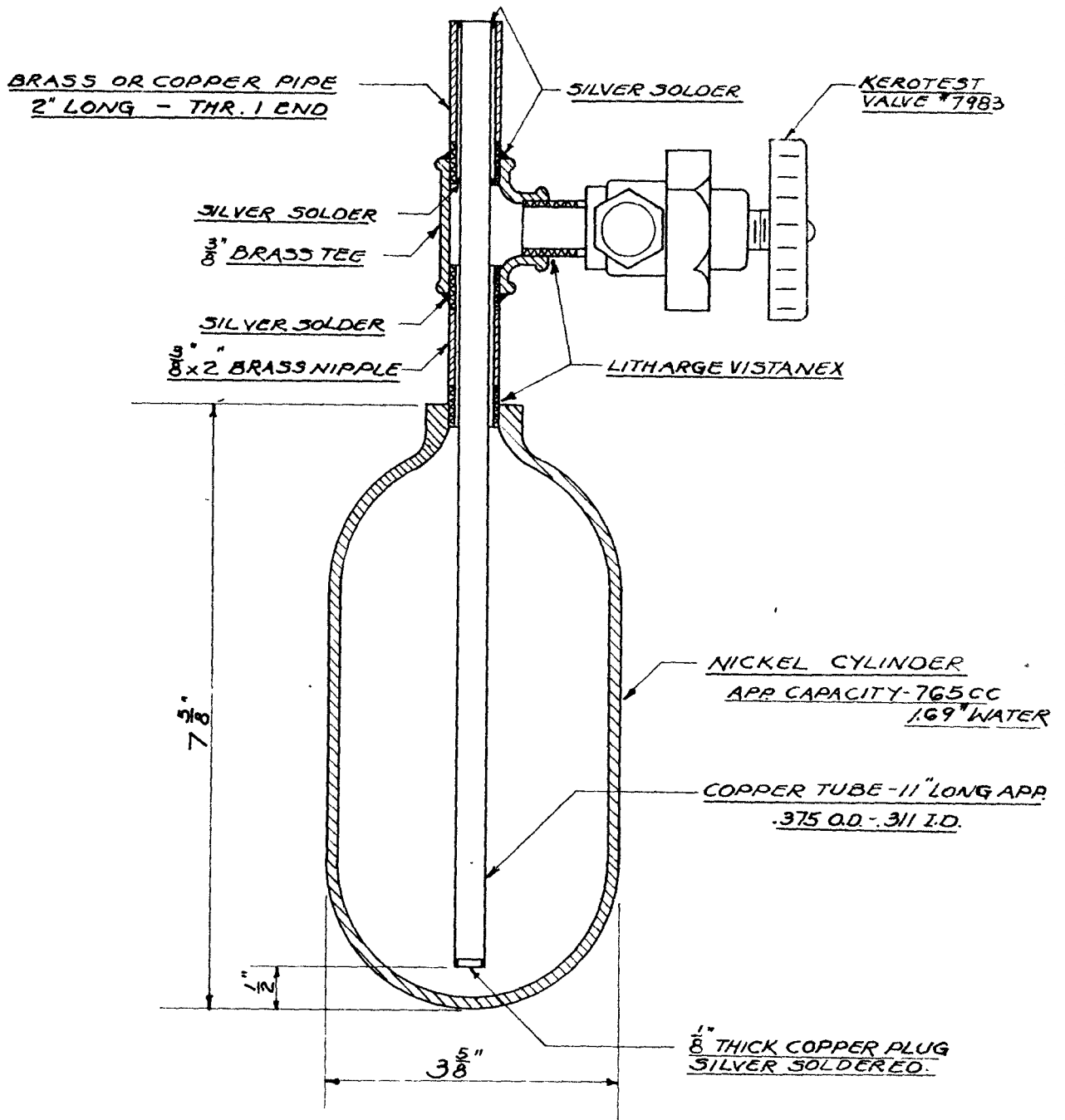
MODIFIED FREEZING POINT CELL

SCALE 6" = 1'-0"

THE HARSHAW CHEMICAL COMPANY
1945 EAST 97TH STREET
CLEVELAND, OHIO

SKETCH NO.
092044

This phenomenon is illustrated by the two curves of Figure 1, both secured using the same sample. It will be noted that in the case when the initial temperature rise was more rapid, a satisfactory freezing point (63.97°C) was secured, whereas with a slower temperature rise, a lower value (63.94°C) was secured. (Note the exaggerated temperature scale which distorts the flat portion of the curve.)

b. Precision of the Freezing Point of a Given Sample

Precision of the freezing point temperature, as would be expected, varies somewhat with the amount of C-616 in the cylinder, the purity, and the rate of crystallization. Variations in temperature during the freezing point period of the curve are, in general, less than those shown in Figure 1. For material freezing at temperatures not lower than about 63.97°C (0.01% HF) variation, in general, was not greater than ±0.002°C. Where slow crystallization occurred, or where small samples were tested, variations approaching ± 0.01°C were encountered. In these instances, it appeared probable that non-uniform cooling, followed by a redistribution of heat, occurred. Under these conditions, it seems debatable whether the highest temperature or the average temperature of the relatively constant portion of the curve should be taken as the freezing point. For practical purposes, the matter is unimportant -- since the difference at most would be 0.01° to 0.02°C.

c. Temperature Scale

When freezing points were first determined using the regular cell, and for early tests using the modified technique, a 10-junction thermocouple of the type specified by Dr. Priest was used. This thermocouple was calibrated by comparison with a resistance thermometer which had been calibrated some considerable time earlier by the Bureau of Standards. Recently, this thermocouple was compared with a new resistance thermometer which had been calibrated by the Bureau of Standards only a few weeks previously. The thermocouple was found to give readings about 0.015°C lower than those secured using the resistance thermometer.

For recent tests the resistance thermometer has been used exclusively. Ice and steam points have been found to check quite closely those secured by the Bureau of Standards and it seems probable that temperature readings secured are accurate to at least ± 0.01°C.

Where comparisons are made for tests involving both thermometer and thermocouple temperature readings, proper corrections have been applied to bring the temperature scales into agreement. In this report temperatures, in general, are rounded off to the nearest hundreth of a degree although readings were taken to thousandths. In many instances, this may result in check values being recorded as differing by 0.01°C, whereas the actual difference was only a few thousandths of a degree.
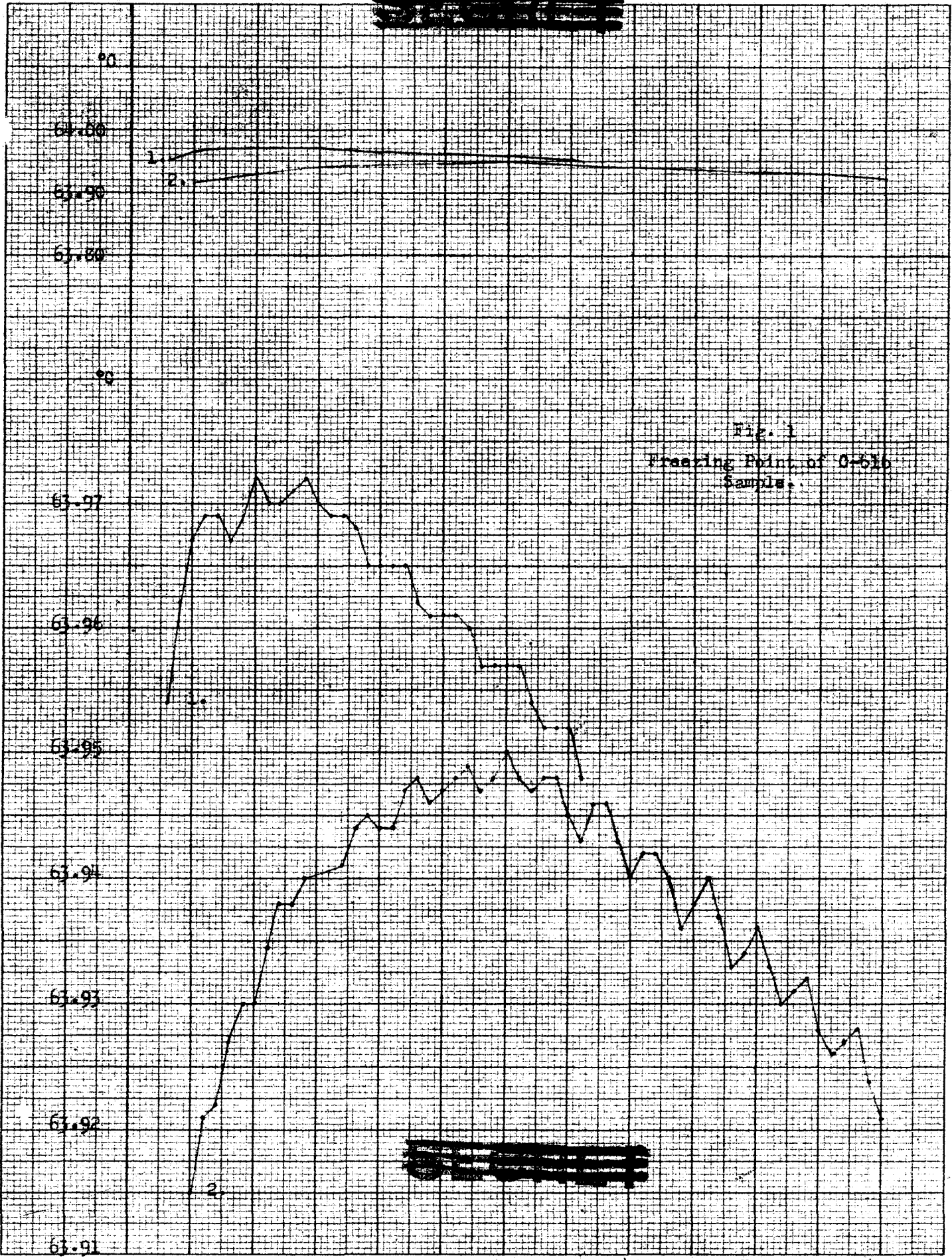
64.00

63.99

63.98

63.97

63.96

63.95

63.94

63.93

63.92

63.91

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Fig. 1

Freezing Point of C-616
Sample.

1

2

10   20   30   40   50   60

As reported earlier, best results appear to be secured when the cooling bath temperature is between 63.0° and 63.4°C. With lower temperatures the sample which has cooled to bath temperature may not recover satisfactorily during crystallization, and short freezing points or slightly lower results (0.01 to 0.03°C) may be secured.

d. Duplication of Results

Duplicate tests on a given sample, provided bath temperature, rate of crystallization, etc. are satisfactory, will be found to check to 0.01°C in practically all cases. Where samples are taken from the production line one after another, providing duplicate samples as nearly as possible, checks have been secured between the various samples, thus indicating satisfactory cross checking of the method.

COMPOSITE SAMPLE I

	F.P.	% HF Kellex data
Cylinder MD-2057	63.98	.009
" MD-2057	63.98	.009
Cylinder MD-2174	63.98	.009
" MD-2174	63.99	.008

COMPOSITE SAMPLE II

	F.P.	% HF Kellex data
Cylinder MD-2169	64.01	.005
" MD-2169	64.02	.004
Cylinder MD-2131	64.01	.005
" MD-2131	64.02	.004

Three sample cylinders, prepared and tested by Harshaw, were sent to other laboratories for checking by the modified freezing point method. Comparative data available are tabulated below, with Harshaw data corrected to the resistance thermometer temperature scale:

	Harshaw	Columbia
Cylinder MD-2193	63.98 ✿	64.04 (oil bath)
	63.98 ✿	64.05 (oil bath)
	63.98 ✿	64.04 (air bath)
	64.01	64.03 (air bath)
	64.02	64.02 (air bath)
	64.02	64.04 (air bath)

✿(Older results of early tests known to be less reliable than susequent runs.)

Nickel sample cylinders in current use have a safe filling capacity of about 5.3 pounds of C-616 when equipped with the thermometer well assembly. Numerous tests were made to determine the effect on the freezing point of varying amounts of C-616 in the cylinder. For plant run material (0.01% HF or less) removal of C-616 from the cylinder was found not to affect the freezing point within practical limits provided at least about three pounds of material remained in the cylinder. If less than three pounds was present freezing point values became less satisfactory, but down to about two pounds results in most cases were only slightly lower than those previously obtained on the larger amount of C-616. Some typical results are as follows:

Cylinder	Weight of Sample	F.P., °C	% HF Kellex data
MD-2088	3.88	64.01	.005
		64.00	.006
	2.99	63.99	.007
		63.99	.007
MD-2092	3.59	64.01	.005
		64.00	.006
	2.81	64.00	.006
		63.99	.006
MD-2143	5.14	63.99	.007
		63.98	.008
	4.56	64.00	.006
		64.00	.006
MD-2198	3.20	64.00	.006
		64.00	.006
	1.92	< 63.96	>.012
		(no flat period secured)	
MD-2077	3.44	63.99	.007
		63.98	.008
	2.23	63.97	.010
		63.97	.010
MD-2163	3.22	63.98	.008
		63.98	.008
	2.70	63.97	.010
		63.97	.010
MD-2161	4.72	63.98	.008
		63.98	.008
	3.57	63.99	.007

e. Effect of Nitrogen on the Freezing Point

In the course of making comparative tests by the original
freezing point, modified freezing point, and reflux techni-
ques, it became apparent (as will be noted later) that
either of the freezing point methods almost invariably
indicated greater HF content than did the reflux analysis.
Since the reflux method had been proved by adding known
amounts of HF with satisfactory recovery, it seemed probable
that other impurities affecting the freezing point might
be involved.

Previous experience with samples in the reflux column, as
cited in earlier reports, showed that nitrogen present in
C-616 is but slowly evolved even in refluxing. Since
receivers used for holding crude C-616 prior to distillation
are opened to the air to a greater or less extent in breaking
connecting lines,etc., nitrogen from the air,as well as
oxygen, may contaminate the crude C-616. The present purifi-
cation technique in all probability would not eliminate all
of the nitrogen (and oxygen). Further, since nitrogen is used
for flushing various lines in the purification system and
complete evacuation is not secured, nitrogen is doubtless
added in small quantities to the purified C-616. Since the
large shipping cylinders are"pressured" with nitrogen,
considerable of this gas will appear in any samples taken
from shipping containers.

With these points in mind, tests were undertaken to determine
whether addition of nitrogen would affect the freezing point
of C-616. In the first two tests nitrogen was added in the
amount necessary to simulate the "pressuring" technique used
for the shipping cylinders. Tests were made in the order,
and with the results, given below - using the modified freezing
point method:

Cylinder MD-2002	F.P.,°C	%HF Kellex Data
1. Freezing Point Test	63.99	.007
	63.99	.007
2. Nitrogen Added		
3. Freezing Point Test	63.96	.012(after 2 hrs)
	63.95	.015(" 4 ")
	63.95	.015(" 24 ")
	63.95	.015(" 24 ")

4. Reflux Test - .011% Total Gas, .003% HF   .011% N_2 (by differenc

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Cylinder MD-2162	F.P., °C	% HF
1. Freezing Point Test	63.98	.009
	63.97	.010
2. Nitrogen Added		
3. Freezing Point Test	63.95	.013
	63.95	.013

To further test the effect of nitrogen addition, a sample cylinder was filled with C-616 beyond its safe capacity and then pumped at room temperature to remove as much HF and nitrogen as possible. After determining the freezing point of this purified material a small amount of nitrogen was added to simulate a small leak of nitrogen without the "pressuring" effect. Subsequently, the cylinder was "pressured" with nitrogen to 40 lbs. gauge at room temperature. Results are tabulated below:

Cylinder MD-2020	F.P., °C	%HF Kellex Data
1. Freezing Point Test	64.02	.004
	64.02	.004
2. Nitrogen Added		
3. Freezing Point Test	64.00	.006
	63.98	.009
	64.00	.006
	64.00	.006
	64.00	.006
4. More Nitrogen Added		
5. Freezing Point Test	63.96	.012
	63.96	.012
	63.95	.013

As indicated by this test, the lowering of the freezing point by nitrogen may be as much as .06°C which would be equivalent to nearly 0.01% HF. Hence, it is apparent that use of freezing point method as a basis for reporting %HF is open to question for samples where nitrogen may be present. (Nitrogen used in the plant has a dew point of -50°C, and moisture added during the pressuring operation would be negligible.)

In one very unusual plant composite sample check values of 63.87°C were secured for the freezing point. By the reflux method, over 0.05% total gas was found but only 0.009% proved to be actual HF. The value .024% HF, corresponding to the 63.87°C freezing point, is too great to be accounted for by nitrogen alone and is doubtless due to other, as yet, unknown factors.

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f. Effect of Air on the Freezing Point

Valves on three sample cylinders previously tested were opened
to admit air into the cylinder. Freezing points were again
run with results shown below:

Cylinder MD-2086	F.P., °C.	Kellex Data %HF
	64.00	.006
	64.00	.006
Air introduced	63.97	.010
	63.95	.012
Cylinder MD-2106		
	64.00	.006
	64.00	.006
Air introduced	63.99 (3)	.007
	63.99 (6)	.007
Cylinder MD-2100		
	63.99	.007
	63.98	.009
Air introduced	63.98	.009
	63.98	.009

As these tests indicate, the effect of inleaking air on the
freezing point will be variable and, in general, quite small.
(Moisture introduced by entrance of air is negligible.)

g. Effect of Storage

Samples in nickel cylinders tested by the modified freezing point
method after extended periods at 100°C showed no appreciable change
in freezing point, as indicated by the following results:

Cylinder No.	After--days	F.P.	%HF Kellex Data
2002	0	63.96	.011
		63.97	.010
		63.98	.009
	7	63.97	.010
		63.97	.010
	21	63.98	.009
		63.98	.009
	28	63.98	.009
		63.98	.009
2020	0	63.99	.008
		63.99	.008
	5	63.99	.008
		63.99	.008

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4. Comparison of Analytical Methods

From a consideration of results reported above it becomes increas-
ingly apparent that the freezing point method of analysis, using either
the original or the modified technique, will give results which depend
on materials other than HF, as well as HF, which may be present. The
reflux method, on the other hand, can be used to differentiate be-
tween impurities condensible at liquid air temperatures and those not
condensible. Soluble, non-volatile compounds would presumably
affect the freezing point but would not become apparent by the reflux
method.

To further add to this confusion, and possibly aid in explaining
some of the discrepancies, the possibility of variation due to
transfer and sampling technique should be considered. In this
connection, data secured in connection with storage tests of C-616
in iron cylinders are of interest. (See Section III of this paper.)

In these tests cylinders were partly filled with C-616 and, after a
time, samples were taken – first, from the vapor, and then from the
liquid, phase. Using the reflux method, actual HF in the vapor phase
samples was found to be 0.13%, 0.13%, 0.11% and 0.13% on different
tests. For liquid phase samples the apparatus was inadequate to
secure accurate results, since the total gas values corresponding
to the above samples were .002% and .003% for the first two determina-
tions and less than .001% for the last two. While these values are
admittedly very approximate, they do clearly indicate that an
appreciable part of the HF may be present in the vapor phase. This
factor would enter into consideration where transfers of C-616 are
made and, particularly, where only partly-filled containers with
relatively large vapor spaces are involved.

Since the two methods (reflux and freezing point), as shown above,
measure the effect of different impurities and have been subject to
variable errors due to transfer technique, it is not surprising that
exact correlation between the methods has not been secured. That
some degree of correlation does exist is shown by the following
results secured on **plant** composite samples. Freezing point results
were secured using the modified technique on samples taken directly
from the production line. Reflux examples were then taken from the
same cylinder.

Cylinder	Reflux Gas Method		Freezing Point Method	
No.	Total Gas%	Actual HF%	°C	%HF Kellex Data
MD-2061	.003	.003	63.98	.009 ☆
			63.98	.009

☆(Differences in %HF for the same temperature are due to values in
the thousandths of a degree not here reported.)

(Contd from Page 12)

Cylinder No.	Reflux Gas Method		Freezing Point Method	
	Total Gas%	Actual HF%	°C	%HF Kellex Data
MD-2088	.008	.001	64.01	.005
			64.01	.005
			64.01	.005
			64.00	.006
MD-2092	.006	.001	64.01	.005
			64.00	.006
MD-2143	.003	--	63.99	.007
			63.98	.008 ✠
MD-2125	.014	.002	63.99	.008 ✠
			63.99	.008
MD-2198	.006	--	64.00	.006
			64.00	.006
MD-2077	.015	.001	63.99	.007
			63.98	.008 ✠
MD-2163	.003	--	63.98	.009 ✠
			63.98	.009
MD-2161	.002	--	63.98	.009
			63.98	.009
MD-2170	.014	.003	63.93	.015
			63.91	.018
			63.93	.015
MD-2132	Over .05	.009	63.87	.024
			63.87	.024

✠ (Differences in % HF for the same temperature are due to values in the thousandths of a degree not here reported.)

II. PRESSURING WITH NITROGEN

Studies of the effect of nitrogen and air on the freezing point of C-616, discussed in a previous section of this paper, have raised questions relative to the merits of pressuring shipping cylinders with nitrogen. Since the prime purpose of introducing nitrogen is to prevent in-leakage of air, an evaluation of the effects of such leakage is pertinent.

Air entering the cylinder well, for practical purposes, contains nitrogen, oxygen and moisture. Nitrogen and oxygen are apparently not detrimental in C-616 since nitrogen has been deliberately added, and no specifications have been set relating to oxygen content. Thus, entrance of air is to be prevented only because of possible contamination due to moisture, and this compound will be given further consideration.

Nickel shipping cylinders in current use have a safe (90% at 100°C) filling capacity of 462 pounds of C-616, which represents approximately 63% capacity at temperatures below the freezing point. In plant practice, cylinders are normally filled to 450 to 455 pounds, and no cylinder contains as little as 425 pounds, which corresponds to approximately 58% solid C-616 in the cylinder. A cylinder containing only 425 pounds of C-616 would contain 40 liters of solid, leaving 28 liters of vapor space, since the volume of the cylinder is approximately 68 liters. At 25°C (77°F) a liter of air saturated with moisture contains approximately 0.023 grams of water, and 28 liters would contain approximately .65 grams of water.

Assume now that the cylinder of C-616, unpressured by nitrogen, developed a leak and the full 28 liters of gas space was filled with saturated air. Thus, .65 grams of water would enter the cylinder. This .65 gram of water would correspond to 0.00034% water in the 425 pounds of C-616 in the cylinder. If this water were to be completely converted to HF, the HF content of the cylinder would be increased by about .00075%, a negligible amount - having less effect on the freezing point than air or nitrogen.

The quantity of moisture added would, in any normal leakage, be even less than that above calculated since the air would not be completely saturated and the gas space in the cylinder would be less than the figure used above.

Thus, pressuring would be of practical value only if a leak developed when the cylinder was submerged in water, or if extremely high humidities were encountered at temperatures considerably above normal where the absolute humidity would be high. In the latter instance, however, the increased vapor pressure of the C-616 at the higher temperature would decrease the available vapor space into which air could leak.

From a production angle, damage due to leakage under such detrimental conditions is excluded since the only point at which high humidities and high temperatures are encountered is at the filling stations where the cylinders are under pressure and any leakage would result in loss of C-616 rather than intake of air.

As stated by C. F. Swinehart in a report covering specifications for cleaning and filling C-616 containers (March 10, 1944), pressuring with nitrogen introduces hazards in heating the cylinders for removal of the C-616.

From a manufacturing standpoint, pressuring with nitrogen involves several time-consuming operations. When the desired weight of C-616 has been charged into the shipping cylinder, the filling line is closed and the pressure on the cylinder is determined in order to know how much nitrogen should be added. This pressure, under normal filling routine, is about 5 to 10 pounds gauge, and nitrogen is added to give about 25 to 30 pounds gauge pressure.

Unfortunately, uniform pressures are not secured on cooling to room temperature and, in order to meet the specification of 5 to 10 pounds gauge pressure at 20°C, each cylinder must be tested individually prior to shipment. Pressures found on test have varied from 25 inches of vacuum to about 25 pounds pressure, and adjustment by adding nitrogen or bleeding off vapor has been necessary.

Since pressure in a cylinder into which air leaked would not exceed atmospheric pressure, less hazard would be encountered than with nitrogen pressuring where the present maximum is 10 pounds higher than atmospheric.

Thus, it would appear that pressuring with nitrogen is of no practical value in excluding moisture as far as production is concerned, and introduces unnecessary hazards and expense. For these reasons, it is suggested that pressuring of the cylinders with nitrogen be discontinued unless the use of nitrogen is required by storage or unloading conditions.

III. STORAGE OF C-616 IN CYLINDERS

At the request of Captain W. M. Hearon, C-616 was charged into two steel cylinders to determine corrosion characteristics which might result. One cylinder, referred to as the "hot" sample, was held at 100°C continuously, while the "cold" sample was kept at room temperature except when samples were taken.

Since any reaction occurring might affect the iron and HF content of the C-616, tests were made to determine these constituents in samples secured from both the liquid and vapor phase of the C-616 in the cylinder.

Liquid phase samples were secured by the usual method for reflux gas and iron determinations as described in Technical Report (Contract No. W-7405 eng. 43) dated August 5, 1944.

Vapor phase samples for the first reflux tests were secured using a gas pipette mounted in the heated box containing the manifold and the heated cylinder. For later reflux tests, and for iron determinations, C-616 was distilled from the cylinder and condensed in the platinum weighing tube attached to the manifold.

Results of the HF determinations are as follows:

	Hot Sample	Cold Sample
	% Actual HF	
Vapor phase · original value	.13	.13
" " – after 6-1/2 wks.	.11	.13

	Hot Sample	Cold Sample
	% Total Gas as HF	
Liquid phase – original value	.0017	.0034
" " – after 6-1/2 wks.	.0007	.0003

Since the apparatus is not designed to measure accurately - impurities in C-616 in the small quantities found (0.5 to 3 milligrams as HF in a 140-gram sample), and the variation might be due to manipulative errors, no definite significance can be attached to the apparent drop in gas content of the liquid phase samples. The decrease in the gas phase HF content, though perhaps partially due to experimental error, may be related to the increase in iron content described below.

As described in an earlier section of this report, some difficulties in securing exact values have been encountered when analyzing for Fe in C-616 using the standard colorimetric method which has proved satisfactory in the analysis of Green Salt (WE-22).

Results as secured are as follows:

	Hot Sample	Cold Sample
	p.p.m. Fe	
Liquid phase – original value	13	36
" " – after 6-1/2 wks.	477	26
Vapor phase – original value	38	17
" " – after 6-1/2 wks.	13	9

Because of analytical difficulties, these values should be considered as approximations only. Thus, the iron content of the cold sample has remained essentially unchanged, while the hot sample (in the liquid phase only, as expected) has increased from a few p.p.m. to almost 500 (0.05%). These results thus indicate no measurable attack at room temperature, while at 100°C a definite, but not serious corrosion, has occurred over the 6-1/2-week period. This corrosion (0.05%), which may be due in part to attack of oxide film on the surface of the cylinder, corresponds to reaction of about 1 gram of iron in the cylinder which has about 60 square inches of surface in contact with Liquid C-616, and about 180 square inches in contact with the vapor.

IV. ANALYSIS OF BY-PRODUCT HF

The second sample of by-product anhydrous HF received from The Hooker Electrochemical Company was analyzed and reported September 6, 1944.