File

DEPARTMENT OF MINES AND RESOURCES

BUREAU OF MINES

CANADA

Ottawa, October 5, 1946.

R E P O R T

of the

ORE DRESSING AND METALLURGICAL LABORATORIES.

Investigation No. 2119.

Metallurgical Examination of a No. 2 Catalyst
Steel Column used for Ammonia Synthesis.

PART I. - Mechanical Testing and Chemical
Analysis.

(Copy No. 12.)

Bureau of Mines
Division of Metallic
Minerals

Physical Metallurgy
Research Laboratories

CANADA

DEPARTMENT
OF
MINES AND RESOURCES

O T T A W A   October 5, 1946.

R E P O R T

of the

ORE DRESSING AND METALLURGICAL LABORATORIES.

Investigation No. 2119.

Metallurgical Examination of a No. 2 Catalyst Steel Column Used for Ammonia Synthesis.

PART I. - Mechanical Testing and Chemical Analysis.

===========

Origin of Material and Object of Investigation:

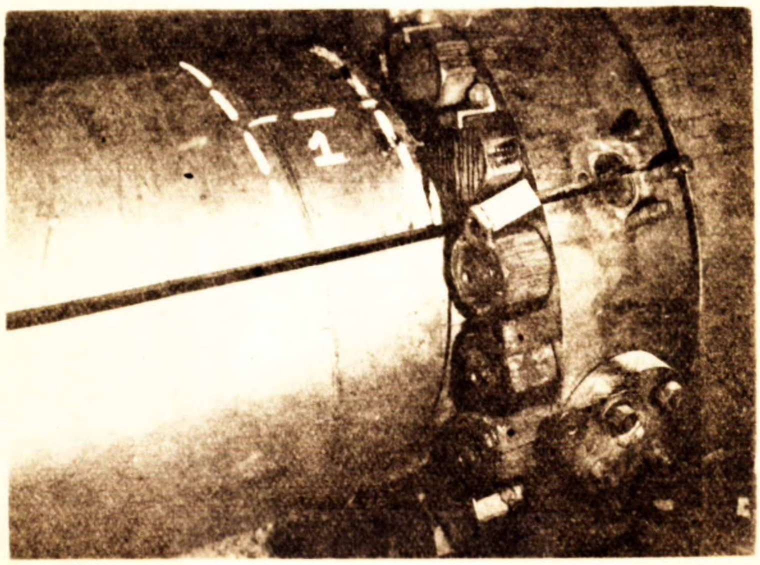
In March, 1946, in the course of a visit, Mr. A. H. W. Busby, Superintendent of Physical Research, The Consolidated Mining and Smelting Company of Canada Limited, Trail, British Columbia, requested a complete mechanical and metallurgical examination of a No. 2 Catalyst steel column which had failed in service while being used in the synthesis of ammonia. During the period from April to July, further information was supplied by the company on the service conditions encountered by the column.

The present report, Part I of this investigation, covers the mechanical testing and the chemical analyses of samples cut from the column.

Material Received:

The company submitted three sections of steel, approximately 14 inches square and 7 inches thick, taken from the fractured ammonia column. The location of these pieces with respect to the column flange is shown in Figure 4.*

Figure 4.



MAIN BREAK OF AMMONIA COLUMN THROUGH ELECTRICAL LEAD-IN PORT IN FLANGE.

The square shown in Figure 4 as numbered "1" and marked in white was retained by the Company. The next square, blocked out in white but not numbered, was sent for investigation, together with two neighbouring squares. The third square had one edge ragged from the fracture of the column which appeared in the column wall, 180° from the cracked electrical lead-in port, shown also in Figure 4.

The fractured electrical lead-in port is shown in Figure 5. A small section of the diffused copper and steel (marked in Figures 5 and 6) has also been submitted for investigation.

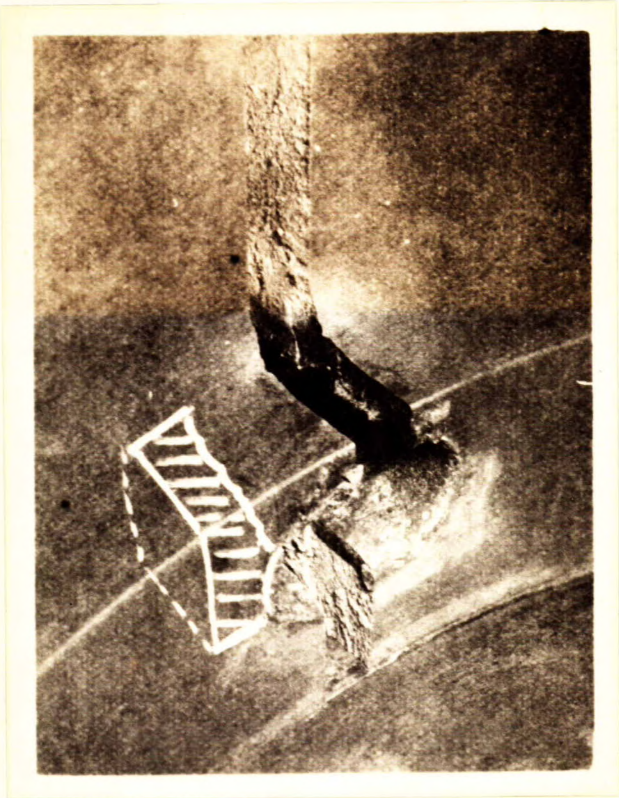
(Figures 5 and 6 follow, on Page 3.)
(Text is continued on Page 4.)

---

* Figures 4, 5 and 6, shown in this report, were made by the Consolidated Mining and Smelting Company of Canada, Limited.

(Material Received, cont'd) -

Figure 5.

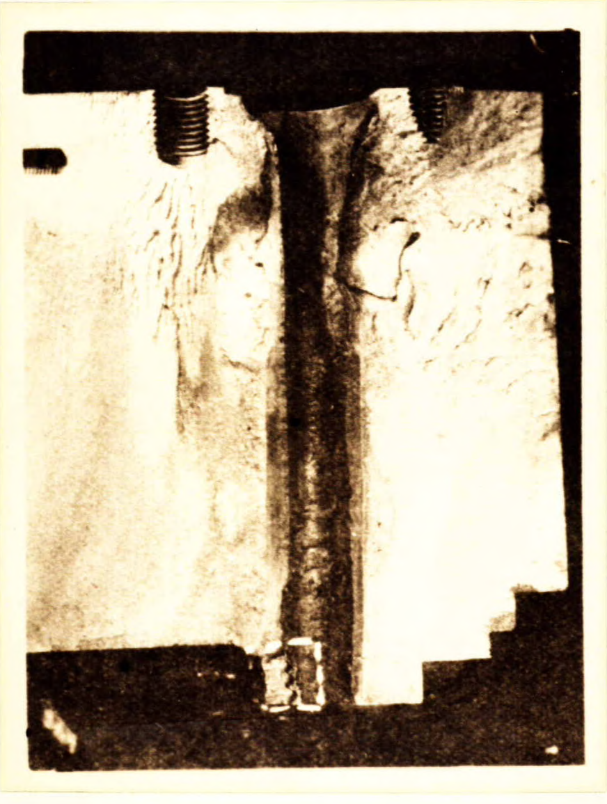


FLANGE AND FRACTURED LEAD-IN PORT,
WITH FUSED COPPER-STEEL ZONE.

Showing the location of sample.

-

Figure 6.



BROKEN SECTION THROUGH ELECTRICAL
LEAD-IN PORT.

Showing location of sample.

-

(Material Received, cont'd) -

Figure 6 shows one-half of the electrical lead-in port after the column had been opened out.

The sample with the diffused copper-steel zone, taken from the column as marked in Figures 5 and 6, has been examined microscopically. Results of that microscopic examination are to be given separately in Part II of this series, Investigation Report No. 2126, dated October 10, 1946.

Description of the Column:

The dimensions of the column, given below, are taken from the Company's Drawing No. CFA-8 showing details of the forging, and Drawing No. CFA-23 showing the original assembly of the complete column.

The principal dimensions of the column are as follows:

Total length = 7,000 mm. (23 feet)

Inside diam. of the middle section = 850 mm. (33.47 inches).

Outside diam. of the flange = 1,670 mm. (65.75 inches).

Inside diam. of the flange = 750 mm. (29.53 inches).

Diam. of the electrical lead-in port = 36 mm. (1.42 inches).

The normal operating pressure during the synthesis of ammonia was 230 atmospheres (3,275 pounds per square inch). The catalyst temperature was about 600° C. The temperature of the steel column did not exceed 220° C., because the steel walls of the column were cooled by the flow of cold inlet gas.

As given by the Company, during the 14 hours of operation prior to the column's being shut down for hydrostatic tests, the temperature of the wall of the forging ranged from 105° C. to 132° C. near the upper flange, and from 201° C. to 218° C. near the lower flange (i.e., near the

(Description of the Column, cont'd) -

mid-joint of the column). The last recorded temperatures after the shut-down and 22 hours before commencing the critical test were 22-25° C. and 60-64° C., for upper and lower flanges respectively.

The temperature difference across the wall of the column is between 20 and 25 degrees C. under normal operating conditions.

The top half of the No. 2 catalyst column failed under hydrostatic pressure at 5,500 pounds per square inch. It is a general practice to test the column at approximately 1.5 times the designed pressure, i.e., 6,600 pounds per square inch.

The design pressure is 4,400 pounds per square inch. The working pressure is about 3,275 pounds per square inch.

The column was forged and was manufactured by the English Steel Corporation Limited, Sheffield, England, in 1931. It had been in continuous operation up to the time of failure, on January 22, 1946.

RESULTS OF INVESTIGATIONS.

Chemical Analysis:

Below are given the chemical analyses obtained by these Laboratories and, for comparison, the analyses of the Consolidated Mining & Smelting Co. and the English Steel Corporation:

(Continued on next page)

(Results of Investigation, cont'd) -

Chemical Analysis (In Per Cent).

		C.M. & S. Testing Division Chemical Analysis 9331	Bureau of Mines Chemical Laboratory			English Steel Corporation
			Outside	Centre	Inside	
Carbon	-	0.31	0.28	0.29	0.30	0.2852-0.30
Silicon	-	0.21	0.20	0.20	0.20	0.226-0.23
Manganese	-	0.65	0.58	0.58	0.58	0.58-0.62
Nickel	-	1.5	1.48	1.51	1.51	1.47-1.49
Chromium	-	0.80	0.73	0.73	0.73	0.86-0.78
Molybdenum	-	0.10	0.06	0.06	0.06	Not reported
Sulphur	-	0.03	0.029	0.035	0.040	" "
Phosphorus	-	0.04	0.029	0.029	0.029	" "

The chemical analysis shows that the steel examined is similar to SAE 3130 steel.

From a comparison of the outside, centre and inside analyses at the Bureau of Mines, only sulphur shows a tendency to segregation (as shown in the results, S = 0.029; 0.035; 0.040).

The chemical analyses determined at the three different laboratories agree closely.

Mechanical Testing of the Column:

Because of the importance of this work, it was decided to perform an extensive mechanical examination of the column.

(a) Tensile Tests -

In the tensile test, 0.505-inch-diameter bars were used. The mechanical properties were determined on samples taken, in the longitudinal and transversal directions, from the outside, centre and inside wall of the column.

The exact positions of the samples are given in the drawings, Figures 1, 2 and 3, placed at the end of this report.

The results obtained from the mechanical tests are given in Table I for the samples taken in the longitudinal

(Mechanical Testing of the Column, cont'd) -

direction, and in Table II for the transverse samples. The results of the impact tests are given in Tables III and IV.

The results of a mechanical examination made on the column in 1931 by the English Steel Corporation Limited[⊕] are compared, below, with those obtained at these Laboratories in the longitudinal and transverse directions. It will be seen that they are substantially the same.

		English Steel Corporation	P. M. R. L.	
			Longi-tudinal Direction (average)	Transverse Direction (average)
Yield Point at 0.1%, p.s.i.	-	51,500-47,000	46,300	46,500
Tensile Strength, p.s.i.	-	94,000-80,000	94,400	93,850
Elongation, per cent	-	27-29	24.1	23.0
Reduction in area, per cent	-	45.9-55.8	50.2	43.5

The average tensile results given in Tables I and II show that the material is homogeneous and that no defects in the steel have been located. The cylindrical surfaces of the samples have not shown any visible discontinuity, cracks, or segregations.

The differences between the samples taken from the outside and inside and also in the longitudinal and transverse directions of the column are such as are to be expected in this kind of forging.

The ratio of the 0.2 per cent proof stress to the ultimate tensile stress is about 50 per cent. It is low, as would be expected for large forgings cooled very slowly from the annealing temperature.

(b) Impact Examination -

The impact properties of the forging have been

[⊕] Information supplied by Consolidated Mining and Smelting Company of Canada, Limited.

(Mechanical Testing of the Column, cont'd) -

investigated thoroughly. The positions of the impact samples in the column are shown in Figures 1, 2 and 3.

The following types of samples have been used:

Izod V-Notch:

Dimensions of sample - 10 x 10 mm.

Notch data - 45° V.; 2 mm. deep; 0.25 mm. root radius.

Charpy V-Notch:

Dimensions of sample - 10 x 10 x 55 mm.

Notch data - 45° V.; 2 mm. deep; 0.25 mm. root radius.

Charpy Keyhole Notch:

Dimensions of sample - 10 x 10 x 55 mm.

Notch data - 5 mm. deep; 1.0 mm. root radius.

The impact values obtained at room temperature are low; for Charpy V-Notch, they amount to 8.9 foot-pounds on the inside of the column for the longitudinal samples and 10.0 foot-pounds for the transverse samples.

(c) High Temperature Investigation -

The high temperature tensile test was performed at 300° C. The results are given in Table V, on Page 16.

In Report No. 98 issued by The Consolidated Mining and Smelting Company of Canada, Limited, Trail, B.C., it is mentioned that the specification called for an elastic limit of 28,000 p.s.i. in the steel at 300° C. The values obtained in these Laboratories were: for 0.1% proof stress, 39,700 and 37,400 p.s.i.; for 0.01% proof stress, 26,000 and 21,500 p.s.i.

The high temperature impact results are given in Table IV, on Page 15. They are much higher as compared with

(Mechanical Testing of the Column, cont'd) -

the room temperature tests.

It is known that for this kind of steel we should expect a low range of impact values at room temperature and a high range at elevated temperatures.

For the kind of steel under consideration, the impact-temperature relation may be divided into three parts:

1. Low level of impact values at lower temperatures, with granular, bright fractures and small deformations.

2. Transition, or scattering, zone with mixed fractures (bright and matte), with impact values between the low and upper levels.

3. Upper level of impact values at elevated temperatures. The fracture is fibrous and matte, and the sample will show more plastic deformation around the notch.

The upper level of the impact value is at the working temperature range. This increases the safety factor of the column.

A study of the influence of temperature on the impact properties, in the range of 0° to 300° C., will form Part III of this investigation series.

A final, fourth report, Part IV, will contain the results of impact tests on small samples, the results of microtensile tests, and a discussion of stress analysis and the safety factor of the failed column.

(d) Dynamic Examination of Material -

Because of the importance of this investigation, stressed by the Consolidated Mining and Smelting Company, fatigue tests were performed on plain and notched samples. For comparison, the endurance limits were determined for this steel under reversed bending loading.

Also for comparison purposes, the endurance limits

(Mechanical Testing of the Column, cont'd) -

for repeated stresses (i.e., for a load range equal to twice the mean load) have been calculated from the results obtained, using the modified Goodman law. The calculated endurance limits for repeated tension loading, namely from 0 to 53,000 p.s.i. for plain samples and 0 to 30,000 p.s.i. for notched samples, mark on a very exaggerated scale the maximum dynamic load which this material can stand.

Dynamic investigations were performed on a large scale because it was generally expected that this material, because of the slow cooling rate from the annealing temperature, might show temper brittleness.

In Table VI are given the results of fatigue tests on samples from the column. Figures 1, 2 and 3 show the locations from which the samples were taken.

The results obtained from the unnotched fatigue samples show a very good homogeneity of the material, since the longitudinal and transverse samples have shown a difference of only 1,000 p.s.i. throughout the material, e.g., 38,000 p.s.i. in longitudinal and 37,000 p.s.i. in transverse direction. It is seen that the material may be loaded dynamically in tension-compression nearly up to 0.01 per cent proof stress without failure.

In tension-compression loading on notched samples, the results were: for the longitudinal direction, inside ±23,000, centre ±22,000, and outside ±23,000 p.s.i.; for the transverse direction, inside ±18,000, centre ±18,000, and outside ±20,000 p.s.i. Here, because of different notch sensitivity, the inhomogeneity was larger than for unnotched material.

The highest stress concentration factor (dynamic notch sensitivity) obtained at these Laboratories was

(Mechanical Testing of the Column, cont'd) -

38,000/18,000, that is, approximately 2, which is comparable with results obtained in other steels of average quality.

(e) Mechanical Examination of Heat-treated Material -

For additional information, the mechanical properties of a section of steel, 1½" x 4" x 7", taken from the column were checked after a special heat treatment.

The heat treatment applied was:

Slow heating up to 840° C.;
Two hours at 840° C.;
Cooled to 400° C. at a rate of 17° C. per hour; and afterwards cooled in the furnace to room temperature.

The results of tensile and impact tests obtained after this heat treatment are given in Table VII.

The values which were obtained for ultimate tensile strength, yield strength, elongation, contraction and impact strength are similar to those obtained on the original material, which indicates that the heat treatment applied was similar to that applied by the English Steel Corporation.

CONCLUSIONS:

As a result of a very extensive mechanical-metallurgical investigation, it was found that the steel used in the fractured column was a good homogeneous material showing rather low impact values at room temperature. The failure originated in the cracking of a fused lead-in port, and was caused by local overheating, melting of the copper-lead, and subsequent cooling of this part.

The discussion on impact properties of this material will be included in the report comprising Part III of this investigation series.

oooooooooooo
oooooooo
oo

(Tables I to VIII and Figures 1 to 3 follow, on Pages 12 to 21.)

TWW:NB:LB.

TABLE I. - Mechanical Properties of Longitudinal Samples, S.A.E. 3130 Steel Ammonia Column.

Location of Specimen	Ultimate Stress, p.s.i.	0.2 per cent Proof Stress, p.s.i.	0.1 per cent Proof Stress, p.s.i.	0.01 per cent Proof Stress, p.s.i.	Elongation, Per Cent in 2"	Per Cent Reduction in Area	Brinell Hardness Number	Izod V-notch, ft-lb.	Charpy V-notch, ft-lb.	Charpy Keyhole Notch, p.s.i.
Outside	93,000	46,500	45,500	44,000	27.0	53.0	179	(15.8 (16.5	16	19
Outside	92,100	46,500	44,800	43,000	25.0	53.0	174	12.3	11	18
Outside	99,500	52,700	49,000	28,000	21.0	40.5	183	13.8	17	20
Average	94,900	48,600	46,400	38,300	24.7	48.8	179	14.6	14.7	19
Centre	93,100	46,800	45,400	41,000	21.0	53.0	174	(12.3 (14.5	16	17
Centre	93,500	47,300	45,700	43,000	25.5	52.0	179	13.8	12	14
Centre	96,500	49,500	47,000	37,000	23.0	47.5	179	11.3	18	14
Average	94,400	47,900	46,000	40,300	23.2	50.8	177	12.9	15.3	15
Inside	92,700	47,200	45,700	43,500	25.5	53.8	179	(10.5 (10.8	7	14
Inside	92,300	46,500	45,700	41,000	25.5	52.5	179	9.5	11	17
Inside	96,800	50,000	47,700	41,000	22.5	47.0	179	11.8	8	16
Average	93,900	47,900	46,400	41,800	24.5	51.1	179	10.7	8.9	15.7

TABLE II. - Mechanical Properties of Transverse Samples, S.A.E. 3130 Steel Ammonia Column.

Location of Specimen	Ultimate Stress, p.s.i.	0.2 per cent Proof Stress, p.s.i.	0.1 per cent Proof Stress, p.s.i.	0.01 per cent Proof Stress, p.s.i.	Elongation, Per Cent in 2"	Per Cent Reduction in Area	Brinell Hardness Number	Izod V-notch, ft-lb.	Charpy V-notch, ft-lb.	Charpy Keyhole notch, ft-lb.
O.W.	91,900	49,000	47,000	45,000	24.0	44.0	174	14.3	18	14
O.W.	92,000	46,500	45,000	44,000	23.0	45.0	174	16.3	18	10
O.W.	96,700	49,500	47,500	42,000	22.0	43.5	179	15.0	13	14
Average	93,500	48,400	46,500	43,700	23.0	44.2	176	15.2	16.3	12.7
Centre	92,100	46,300	45,000	44,000	23.0	45.0	174	10.0	14	15
Centre	93,000	46,200	44,900	42,000	23.0	36.0	174	9.3	14	16
Centre	96,000	49,600	47,500	41,900	21.0	45.0	179	11.3	8	17
Average	93,700	48,400	46,500	42,600	22.3	42.5	176	10.2	12	16
I.W.	93,000	46,300	44,900	44,000	23.0	45.0	170	10.0	12	12
I.W.	93,300	45,900	45,900	44,000	24.5	46.0	174	9.3	11	12
I.W.	97,000	50,700	48,600	44,000	24.0	40.5	170	11.3	7	15
Average	94,400	47,600	46,500	44,000	23.8	43.8	171	10.2	10	13

O.W. = Outside wall. I.W. = Inside wall.

TABLE III. - Impact Properties of S.A.E. 3130 Steel Ammonia Column. (In Izod foot-pounds per sample)*

	LONGITUDINAL SAMPLES					TRANSVERSE SAMPLES				
	Notch 1	Notch 2	Notch 3	Notch 4	Aver.	Notch 1	Notch 2	Notch 3	Notch 4	Aver.
O U T S I D E W A L L -										
Sample #1	17	14	16	16	15.8	-	14	20	9	14.3
#2	18	12	18	18	16.5	17	18	18	12	16.3
#3	14	10	13	12	12.3	15	18	12	-	15.0
#4	11	15	13	16	13.8					
Aver.					14.6				Aver.	15.2
C E N T R E										
Sample #1	10	14	9	16	12.3	9	9	9	8	8.8
#2	17	16	13	12	14.5	19	19	20	11	17.3
#3	12	15	13	15	13.8	18	16	16	12	15.5
#4	8	17	8	12	11.3					
Aver.					12.9				Aver.	13.9
I N S I D E W A L L										
Sample #1	8	10	14	10	10.5	9	9	7	15	10.0
#2	10	12	8	13	10.8	10	9	9	9	9.3
#3	8	10	12	8	9.5	11	10	12	12	11.3
#4	10	12	13	12	11.8					
Aver.					10.7				Aver.	10.2

* Dimensions of the sample: 10 x 10 mm.; V Notch, 0.25 mm. root radius, 2 mm. deep.

TABLE IV - Impact Properties of S.A.E. 3130 Steel Ammonia Column. (In Charpy foot-pounds per sample)®

	LONGITUDINAL SAMPLES		TRANSVERSE SAMPLES	
	V-Notch	Keyhole	V-Notch	Keyhole
OUTSIDE WALL				
Sample #1	16	19	18	14
#2	11	18	18	10
#3	17	20	13	14
Aver.	14.7	19	Aver. 16.3	12.7
CENTRE				
Sample #1	16	17	14	15
#2	12	14	8	16
#3	18	14	14	17
Aver.	15.3	15	Aver. 12	16
INSIDE				
Sample #1	7	14	12	12
#2	11	17	11	12
#3	8	16	7	15
Aver.	8.9	15.7	Aver. 10	13

Impact Properties of Longitudinal Sample of Above Column at 300° C. (In Charpy foot-pounds per sample)®

		Outside Wall	Centre	Inside Wall
Sample #1	-	59	53	47
#2	-	63	60	51
#3	-	62	59	47
Average		61.3	57.3	48.3

® Dimensions of Charpy Samples:

V-Notch - 10 x 10 x 55 mm.; notch 2 mm. deep; root radius, 0.25 mm.

Keyhole - 10 x 10 x 55 mm.; notch 5 mm. deep; root radius, 1.0 mm.

TABLE V. - Mechanical Properties, at 300° C., of Longitudinal Samples, S.A.E. 3130 Steel Ammonia Column.

Location of Specimen	Ultimate Stress, p.s.i.	0.2 per cent Proof Stress, p.s.i.	0.1 per cent Proof Stress, p.s.i.	0.01 per cent Proof Stress, p.s.i.	Elongation, Per Cent in 2 inches	Reduction in Area, Per Cent
Outside wall	87,800	43,700	39,700	26,000	21,0	36.0
Centre	86,800	42,500	37,400	21,500	21.0	38,5

TABLE VI. - Fatigue Tests Using Moore Rotating Beam Fatigue Machines (10,000 r.p.m.).

Stress, ±p.s.i.	Cycles to Failure: A (inside)	B (centre)	C (outside)
"A" Unnotched Longitudinal Samples* -			
50,000	252,000	-	-
45,000	2,271,000	737,000	-
42,000	-	1,870,000	1,622,000
40,000	-	2,948,000	1,877,000
39,000	10,568,000	5,121,000	5,431,000
38,000	$+ 10^7$	$+ 10^7$	$+10^7$
"A" Unnotched Transverse Samples* -			
45,000	249,000	408,000	438,000
40,000	1,986,000	1,073,000	920,000
39,000	-	1,416,000	3,282,000
38,000	2,782,000	$+ 10^7$	6,378,000
37,500	-		5,317,000
37,000	$+ 10^7$		$+ 10^7$
36,000	$+ 10^7$		
"B" Notched Longitudinal Samples** -			
25,000	339,000	-	-
25,000	605,000	-	-
24,000	837,000	463,000	837,000
23,000	$+ 10^7$ ≠	410,000	$+ 10^7$
22,000	$+ 10^7$	$+ 10^7$	$+ 10^7$
20,000	$+ 10^7$	$+ 10^7$	
"B" Notched Transverse Samples** -			
25,000	310,000	-	-
23,000	484,000	-	-
22,000	-	2,774,000	522,000
21,000	1,227,000	-	-
20,000	2,774,000	3,547,000	$+ 10^7$
19,000	2,009,000	4,552,000	-
18,000	$+ 10^7$	$+ 10^7$	$+ 10^7$
17,000	-	$+ 10^7$	
16,000	$+ 10^7$		

* Standard 0.2-in.-diam. specimen.

** 0.4-in.-diam. specimen with 0.05-in.-radius notch.

≠ No break.

TABLE VII. - Summary of Endurance Test Results on Samples from S.A.E. 3130 Steel Ammonia Column.

(Tested to 10^7 cycles.)

Section	Location	Stress, ±p.s.i. Unnotched Sample*	Notched Sample**
Longitudinal	Outside wall	38,000	23,000
"	Centre	38,000	22,000
"	Inside wall	38,000	23,000
Transverse	Outside wall	37,000	18,000
"	Centre	38,000	18,000
"	Inside wall	37,000	20,000

* Standard R.R. Moore 0.300-in. diameter.

** 0.400-in. diameter with 0.050-in.-radius notch.

TABLE VIII. - Mechanical Properties of S.A.E. 3130 Steel Ammonia Column After Heat Treatment.

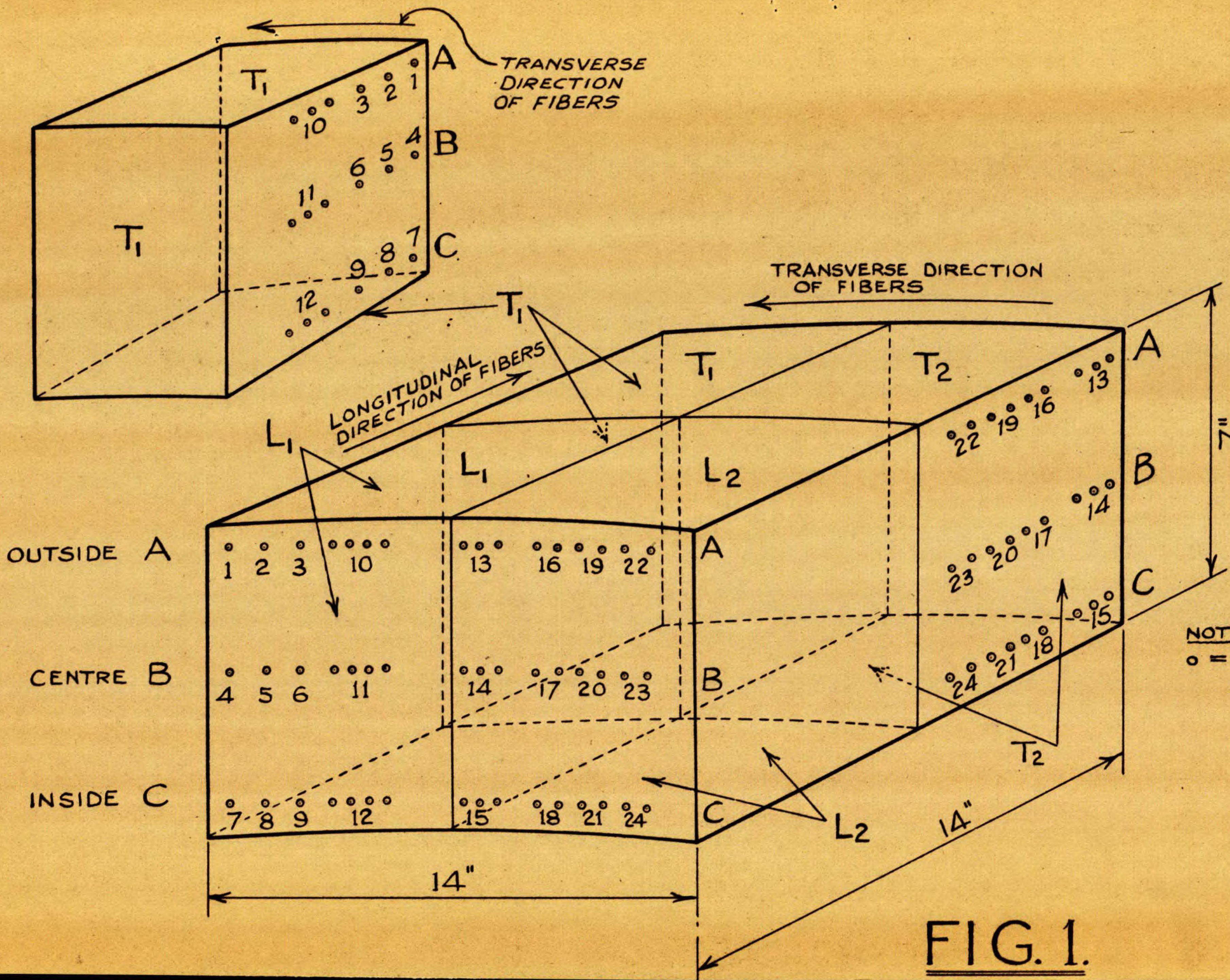
	Sample No. 1	Sample No. 2
Ultimate Stress, p.s.i.	90,300	90,300
Yield Stress, p.s.i.	46,300	46,500
Elongation, per cent in 2 inches	24.0	24.5
Per Cent Reduction in Area	45.0	49.0
Brinell (3,000-kg. load)	170	170

Impact Tests, Charpy foot-pounds:

	Keyhole Notch	"V" Notch
	17	13
	18	17
	16	8
	20	14
	18	18
	18	15
Average -	17.8	14.2

(Figures 1 to 3 follow, on Pages 19 to 21.)

TWW:NB:LB.

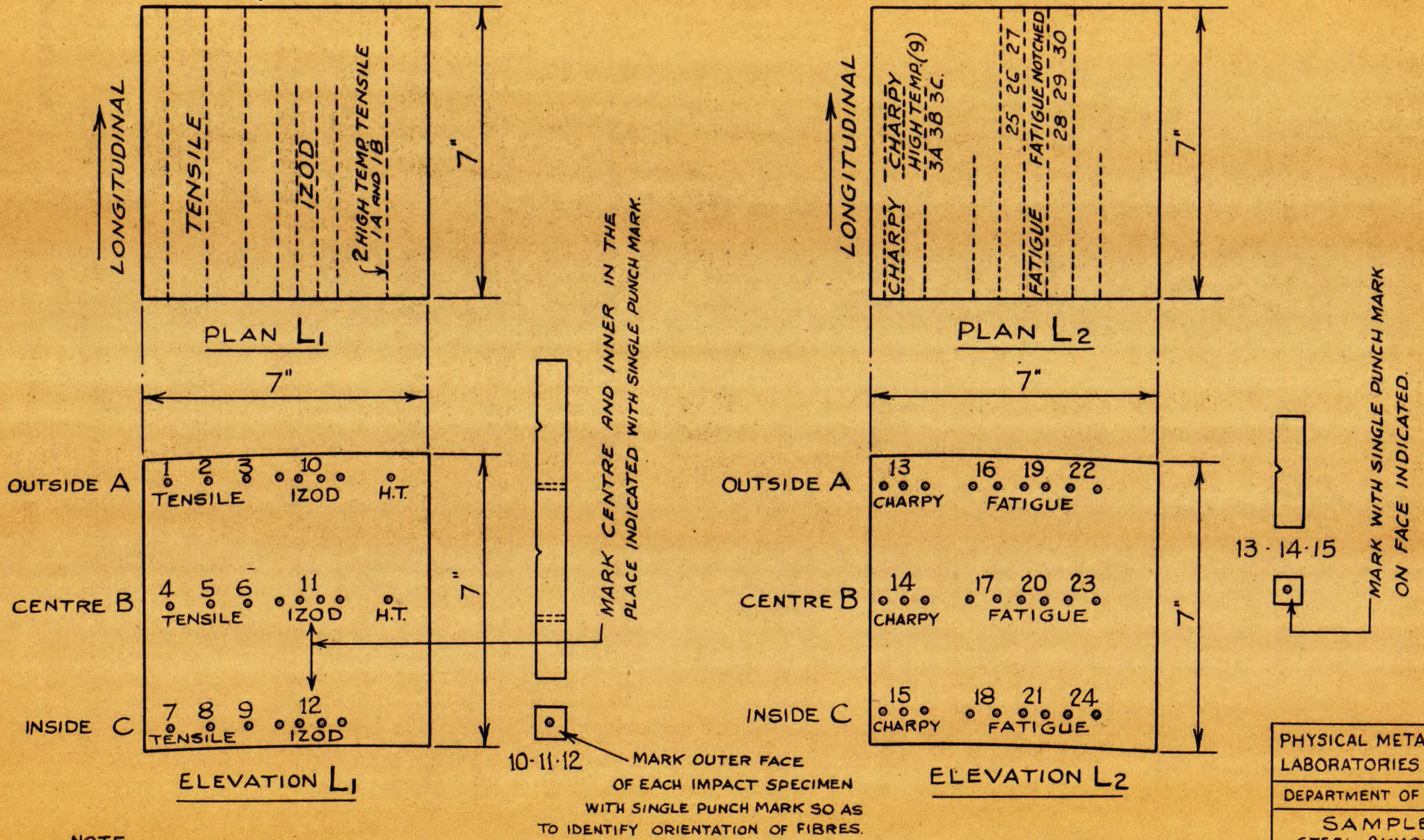


NOTE
o = FACE END OF HORIZONTAL AXIS OF SAMPLE

NOT TO SCALE
DATE 18.7.46.

PHYSICAL METALLURGY RESEARCH LABORATORIES BOOTH ST. OTTAWA
DEPARTMENT OF MINES & RESOURCES
SAMPLES FROM STEEL AMMONIA COLUMN
CHECKED N.B. DRAWN W.A.E. TRACED W.A.E.

FIG. 1.



NOTE

- - - - - - - - - - = HORIZONTAL AXIS OF SAMPLE

o = FACE END OF HORIZONTAL AXIS OF SAMPLE

FIG. 2.

PHYSICAL METALLURGY RESEARCH LABORATORIES BOOTH ST. OTTAWA		
DEPARTMENT OF MINES & RESOURCES		
SAMPLES FROM STEEL AMMONIA COLUMN		
DESIGNED	DRAWN W.A.E.	TRACED W.A.E.
SCALE NONE DATE 18·7·46	APPROVED	CHECKED N.B.

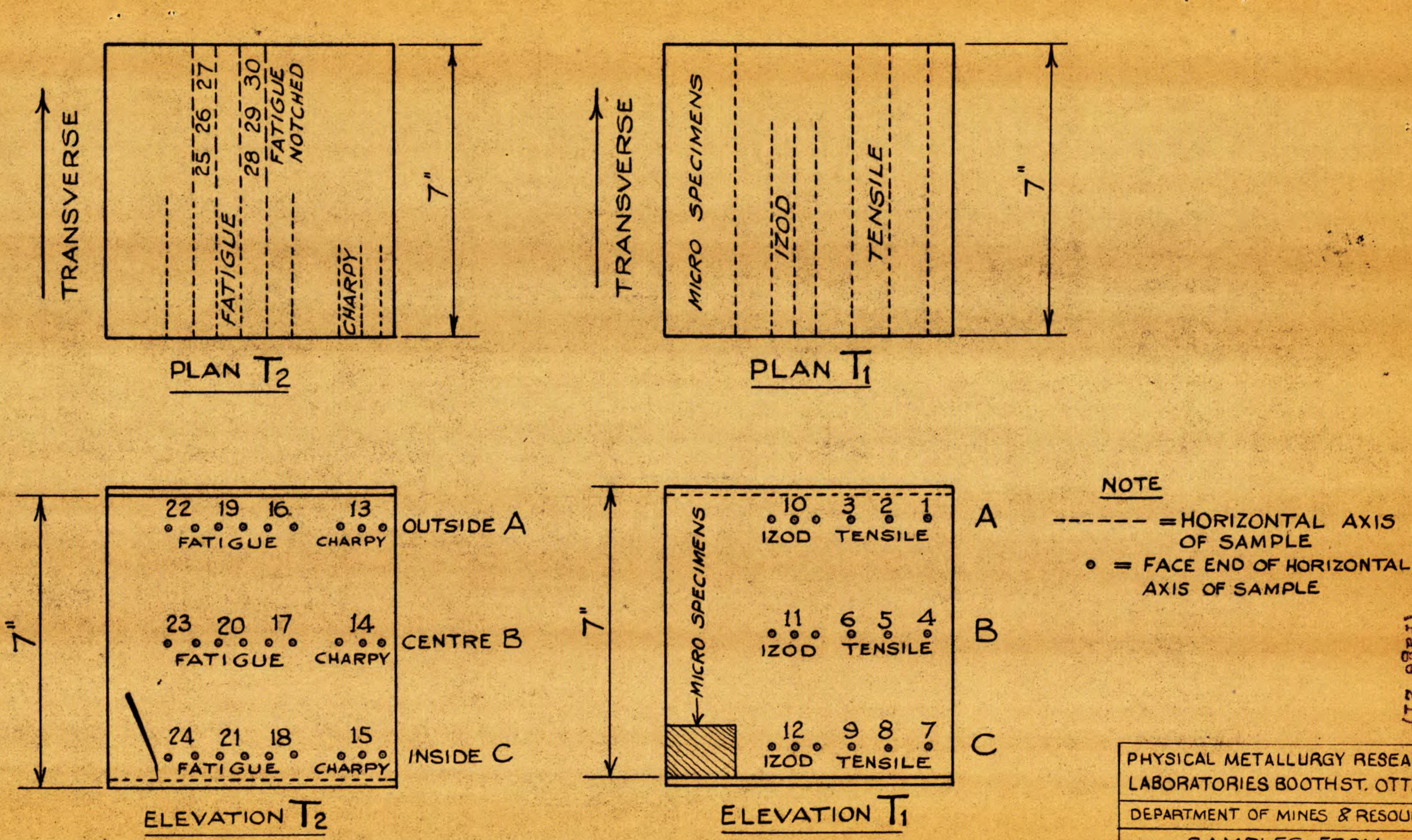


FIG. 3.

PHYSICAL METALLURGY RESEARCH LABORATORIES BOOTH ST. OTTAWA
DEPARTMENT OF MINES & RESOURCES
SAMPLES FROM STEEL AMMONIA COLUMN
DESIGNED DRAWN W.A.E. TRACED W.A.E.
SCALE NONE DATE 18.7.46 APPROVED CHECKED N.B.

JAW : NB : LB.