

NOTEBOOK NO. 1  
ISSUED TO Lathin Koutchou  
ON 8/19 2005  
DEPARTMENT Lab 206  
RETURNED 1/3 2008

SCIENTIFIC NOTEBOOK COMPANY  
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STEVENSVILLE, MICHIGAN 49127  
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## TITLE

Date	Item	Activity
30 Jan, 06	INSTRON	Measuring energy at break for ALCAN pouches
2 Feb, 06	INSTRON	Measuring energy at break for TOPAN pouches
8 Feb, 06	INSTRON	Measuring energy at break for MKC pouches
9 Feb, 06	INSTRON	Measurement of seal strength and energy at break
14 Feb, 06	INSTRON	Measurement of seal strength
25, MAY 2006	OMEGA	Welding thermocouple
25 MAY 2006	OMEGA	Calibration

Sign and date after each entry.

## TITLE

Experiment: Effect of packaging material on pre-heating time and packaging integrity after HPTS processing planning

### CORANET STP 2015 – Phase II

STP-2015 Project Shelf Stable Egg-Based Products Processed By Ultra High Pressure Technology

#### Egg patty size for "institutional" pouches



Pouch net weight: 6 oz (170 g)  
Pouch size (egg patties): 3" x 6" x 0.5"

Formulation: 20% cheese

Runs: 19

#### Conditions

HPTS1: 75°C/700 MPa, 121°C, 3 min: **9 runs**  
HPTS2: 90°C 700 MPa, 105°C, 5 min: **10 runs**

Sign and date after each entry.

## TITLE

## 1. HP System characterization

### *Objective:*

Location of coldest point within the carrier

### *Materials and Methods:*

Determine temperature distribution within the carrier with various thermocouples.

Locate pouch with two thermocouples at the coldest point in the carrier (probably carrier at the wall). One thermocouple goes outside the pouch against the carrier wall and the other in the center of the pouch.

This should show if there are temperature gradients during the process.

**Carrier inside diameter:** 4.7 inches (120 mm)

**Carrier length:** 35.8 inches (910mm)

**Nominal height per shelf:** 7.8 inches (200 mm).

**Number of shelves:** 4 (Each shelf will hold 2 to 3 pouches)

The shelves will be designed to hold the pouches with space around each pouch for circulation in the cooker. This carrier volume needs to be verified by the temperature mapping. As such, these dimensions (120 mm dia x 910 mm long) represent the maximum estimated product volume of **10.3 L**.

**Pouches per load:** 10 per load

## 2. Formulation verification and scale up (WSU, NCFST, OSU, Avure)

### 2.1 Comparison between machines

#### *Objective:*

1. To compare differences in final quality when egg patties in institutional pouches are treated in large or small vessels
2. Evaluate the effect of one stage and two-stage preheating method

#### *Experimental design*

2x2 Factorial: [preheated 75°C, preheated two stage {(i) 60°C (ii) 75°C}] x [HPTS1 Avure 35 L, HPTS1 OSU 1.5 L]

2 replicates

HPTS1– 700 MPa, 105°C, 5 min using 6 oz pouches

#### *Materials and Methods*

- Egg patty type: Michael foods (46025-70019-00) 20% cheese: **3 oz**

Two patties per pouch  
Patties needed: **60 (3 oz)**

- Packaging material: ALCAN (Biaxial Nylon / Adhesive / 5.0 ml EVOH/ Coextruded Sealant): **25 pouches**

**Sign and date after each entry.**

## TITLE

- Preheating method selected: Steam / air injection at 100°C
- HPTS processing
  - Avure 35 L: 700 MPa, 105°C, 5 min
  - OSU 1.5 L: 700 MPa, 105°C, 5 min
- Testing: Texture Profile Analysis, Syneresis, Color (L\*, Chrome)
- Location: *HPP processing*: OSU, Avure. *Testing*: WSU

## 2.2 Comparison between individual and institutional egg patties

### Objectives:

1. To determine if formulation with 20% cheese is accepted by consumers after HPTS treatment at 121°C
2. To evaluate acceptability of individual and institutional egg patties after HPTS at 105°C and 121°C compared with conventional retorted trays (121°C).

### Experimental Design

2 x 3 factorial: [1.5 oz, 6 oz] x [preheated 75°C, HPTS1, HPTS2]

2 replicates for analytical quality tests  
1 replicate for consumer panel

HPTS1– 700 MPa, 105°C, 5 min  
HPTS2– 700 MPa, 121°C, 3 min

2-stage preheat: preheat patties up to 60°C, then apply selected preheating method

### Materials and Methods

- Egg patties type:  
Michael foods (46025-70019-00) 20% cheese: 1.5 oz, 3 oz.  
  
For larger size, place two patties per pouch  
  
Patties needed for analytical testing: 60 (1.5 oz), 45 (3 oz)  
Patties needed for consumer testing (40 panelists need 30 oz egg):  
1.5 oz standard (preheated): 25 patties  
1.5 oz cheese (preheated + HPTS2): 50 patties  
3 oz cheese: (preheated + HPTS1 + HPTS2): 75 (3oz)
- Packaging material: ALCAN (Biaxial Nylon / Adhesive / 5.0 ml EVOH/ Coextruded Sealant): 150 pouches
- Preheating method: Steam / air injection at 100°C
- Testing: time-temperature profiles, ( $f_h$ ,  $j_h$ ) values,  $F_0$   
Consumer panels (40 members), texture profile analysis, color (L\*, chrome), syneresis, incubation tests
- Location: *HPTS processing* NCFST or Avure. *Sensory*: WSU

Sign and date after each entry.

TITLE

### 3. Packaging material testing (NCFST, WSU)

#### 3.1 Effect of packaging material on pre-heating time and packaging integrity after HPTS processing

*Objectives:*

1. To understand how packaging materials affect preheating time
2. To understand the effect of preheating on packaging materials integrity and O<sub>2</sub> permeability after HPTS

*Experimental Design*

3 x 2 factorial: [retort pouch, ALCAN type, Topan] x [preheat up to 90°C, HPTS2]  
3 replicates

HPTS2– 700 MPa, 121°C, 3 min

*Materials and Methods*

- Egg patty type: Michael foods (46025-70019-00) 20% cheese: 1.5 oz  
Patties needed: **100 (1.5 oz)**
- Packaging material:  
Retort pouch: Smurfit: **15 pouches**  
ALCAN (Biaxial Nylon / Adhesive / 5.0 ml EVOH/ Coextruded Sealant): **15 pouches**  
Topan: **15 pouches**
- Preheating methods:  
Water bath at 90°C  
Steam / air injection at 100°C
- Testing: time-temperature profiles, ( $f_h$ ,  $j_h$ ) values, Mocon oxtran, packaging integrity (seal, visual)
- Location: NCFST or Avure (WSU)
- Testing date: October

#### 3.2 Effect of residual headspace on packaging integrity after HPTS

*Objectives:* To understand the effect of residual headspace on packaging materials integrity and O<sub>2</sub> permeability after HPTS

*Experimental Design*

3 X 2 factorial: [3 headspace levels] x [control, HPTS2]

3 replicates

HPTS2 – 700 MPa, 121°C, 3 min

Sign and date after each entry.

TITLE

#### 4. Summary: Avure runs

- Preheating methods:

One-stage: starting at room temperature

Two-stage: equilibrate at 60C, preheat at 75C or 90 C

- Pressurization conditions:

HPTS1: 75°C/700 MPa, 121°C, 3 min: **9 runs**

HPTS2: 90°C 700 MPa, 105°C, 5 min: **10 runs**

Note: 10 pouches (6 oz) per cycle

Run #	Preheating	HPTS	Samples	Total samples	Ref. to
1	One-stage	1	2 (6oz) + 3 (6oz TC) + 2 TC free	5	1; 2
2	One-stage	1	2 (6oz) + 3 (6oz TC) + 2 TC free	5	1; 2
3	Two-stage	1	2 (6oz) + 3 (6oz TC) + 2 TC free + 2 (1.5oz)	7	1; 2; 3
4	Two-stage	1	2 (6oz) + 3 (6oz TC) + 2 TC free + 2 (1.5oz)	7	1; 2; 3
5	Two-stage	1	2 (1.5oz) + 7 (6oz) + 1 (6oz TC)	10	3
6	Two-stage	1	2 (1.5oz) + 7 (6oz) + 1 (6oz TC)	10	3
7	Two-stage	2	2 (1.5oz) + 7 (6oz) + 1 (6oz TC)	10	3
8	Two-stage	2	2 (1.5oz) + 7 (6oz) + 1 (6oz TC)	10	3
9	Two-stage	1	9 (6oz) + 1 (6oz TC)	10	3
10	Two-stage	1	9 (6oz) + 1 (6oz TC)	10	3
11	Two-stage	1	9 (1.5oz) + 1 (1.5 oz TC)	10	3
12	Two-stage	1	9 (1.5oz) + 1 (1.5 oz TC)	10	3
13	Two-stage	2	9 (6oz) + 1 (6oz TC)	10	3
14	Two-stage	2	9 (6oz) + 1 (6oz TC)	10	3
15	Two-stage	2	9 (1.5oz) + 1 (1.5 oz TC)	10	3
16	Two-stage	2	9 (1.5oz) + 1 (1.5 oz TC)	10	3
16	Two-stage	2	6 (1.5 oz, dif. packaging) + 2 (1.5 oz., dif. headspc.) + 2 (6oz TC) + 2 TC free	10	1; 4
18	Two-stage	2	6 (1.5 oz, dif. packaging) + 2 (1.5 oz., dif. headspc.) + 2 (6oz TC) + 2 TC free	10	1; 4
19	Two-stage	2	6 (1.5 oz, dif. packaging) + 2 (1.5 oz., dif. headspc.) + 2 (6oz TC) + 2 TC free	10	1; 4

1. Temperature distribution studies
2. Comparison one-stage vs. two-stage preheating
3. Quality and acceptability testing
4. Packaging testing

Sign and date after each entry.

TITLE

ALCAN, Topan AND MRE pouches were tested at Arise November 17, 2005.

Samples were sent back to NCFST and kept in refrigerated conditions.

Experimental design is described below:

## Packaging material testing (NCFST, WSU)

### 1.1. Effect of packaging material on pre-heating time and packaging integrity after HPTS processing

#### Objectives:

1. To understand how packaging materials affect preheating time
2. To understand the effect of preheating on packaging materials integrity and O<sub>2</sub> permeability after HPTS

#### Experimental Design

3 x 2 factorial: [retort pouch, ALCAN type, Topan] x [preheat up to 90°C, HPTS2]

3 replicates

HPTS2- 700 MPa, 121°C, 3 min

#### Materials and methods:

##### Egg patty type:

Michael foods (46025-70019-00) 20% cheese: 1.5 oz

##### Packaging material:

MRE

ALCAN (Biaxial Nylon / Adhesive / 5.0 ml EVOH/ Coextruded Sealant):

Topan:

##### Preheating methods:

Water bath at 90°C

Steam / air injection at 90°C

##### Testing:

time-temperature profiles, (f<sub>h</sub>, j<sub>h</sub>) values, Mocon oxtran, packaging integrity (seal (Instron), visual)

Sign and date after each entry.



TITLE

ALCAN peeling setup by INSTRON

Sample ID	ALCAN
Test Type	Tensile
Number of specimens	15
Geometry	Rectangular
Test Date	30-Jan-06
Operator Name	Ilona
Units	Metric
Machine type	4200/4300/4400
Custom Test Label	
Data Rate (pts/sec)	6.667
Second Data Rate (pts/sec)	0
Crosshead Speed (mm/min)	10
Second Speed (mm/min)	0
Third Speed (mm/min)	0
Temperature (Deg F)	73
Humidity (%)	50
Sample comments	
Series IX version	8.31.01
Method	50

	Width (mm)	Thickness (mm)	Spec gauge len (mm)	Ext. gauge len (mm)
CONTROL	24.5	0.29218	110	190
CONTROL	24.5	0.29218	110	190
HPP	24.5	0.29218	110	190
HPP	24.5	0.29218	110	190
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HPP	24.5	0.29218	110	190
HPP	24.5	0.29218	110	190
HPP	24.5	0.29218	110	190
PREHEATING	24.5	0.29218	110	190
PREHEATING	24.5	0.29218	110	190
MIN VACUUM	24.5	0.29218	110	190
MIN VACUUM	24.5	0.29218	110	190
MIN VACUUM	24.5	0.29218	110	190
MED VACUUM	24.5	0.29218	110	190
MED VACUUM	24.5	0.29218	110	190
MED VACUUM	24.5	0.29218	110	190
MIN PREHEATING	24.5	0.29218	110	190
MIN PREHEATING	24.5	0.29218	110	190
MIN PREHEATING	24.5	0.29218	110	190
MED PREHEATING	24.5	0.29218	110	190
MED PREHEATING	24.5	0.29218	110	190
MED PREHEATING	24.5	0.29218	110	190

Sign and date after each entry.

	Load at Peak (kgf)	Displcment at Peak (mm)	Displcment at Break (mm)	Displcment at 0.2% Yield (mm)	% Strain at 0.2% Yield (%)	Young's Modulus (kgf/mm <sup>2</sup> )	Energy at Yield (kgf-mm)	Energy at Break (kgf-mm)
Control	9.725	82.55	82.88	3.6403	3.30936	26.4314	8.98171	657.553
Control	9.076	70.26	70.72	4.5137	4.10336	23.088	10.5502	528.538
Mean	9.4005	76.405	76.8	4.077	3.70636	24.7597	9.76595	593.0455
HPP	9.503	61.1	62.4	4.3396	3.94509	24.8262	11.6064	506.535
HPP	9.203	56.07	56.6	3.9856	3.62327	25.9384	10.6152	446.618
HPP	8.674	59.04	59.83	4.1857	3.80518	26.81	10.0861	443.121
HPP	8.966	58.97	60.27	3.8606	3.50964	23.2055	9.76902	456.667
HPP	9.436	61.51	63.18	4.0117	3.647	28.852	10.4593	506.603
Mean	9.201	60.24	61.725	3.93615	3.57832	26.02875	10.1141	481.635
Preheating	8.625	37.02	41.88	3.958	3.59818	23.3282	10.4478	297.647
Preheating	9.611	69.38	70.1	5.062	4.60182	26.9134	11.3317	563.275
Mean	9.118	53.2	55.99	4.51	4.1	25.1208	10.88975	430.461
Min vac	9.262	61.33	62.73	4.4077	4.007	25.9915	10.622	490.539
Min vac	9.377	55.15	55.8	3.6657	3.33245	28.4948	10.0527	447.958
Min vac	9.577	63.57	64.69	4.0357	3.66882	29.1041	10.4346	526.792
Mean	9.405333	60.01667	61.07333	4.036367	3.669423	27.86346	10.36977	488.42966
Med vac	9.117	56.48	57.12	5.212	4.73818	24.4012	10.4087	432.297
Med vac	8.854	55.45	56.05	4.6387	4.217	23.7541	10.1021	417.51
Med vac	8.939	65.4	65.77	4.6143	4.19482	22.2708	10.7504	496.032
Mean	8.97	59.11	59.6466	4.821667	4.38333	23.47536	10.4204	448.613
Min preheat	9.149	54.22	54.35	3.2857	2.987	32.1834	9.39139	430.097
Min preheat	9.35	63.17	63.44	3.8639	3.51264	27.2361	10.4177	503.621
Min preheat	8.829	45.43	45.97	4.015	3.65	26.7663	10.2029	348.15
Mean	9.109333	54.27333	54.5866	3.721533	3.38321	28.7286	10.004	427.28933
Med preheat	9.192	72.94	74.01	10.337	4.45518	14.7325	18.4998	450.231
Med preheat	9.637	56.81	57.34	5.0107	4.55518	31.2445	10.6981	463.65
Med preheat	9.778	57.82	58.58	4.1587	3.78064	32.0606	10.5768	426.234
Mean	9.535667	62.52333	63.31	6.502133	4.16791	26.012	13.25823	446.705

Sign and date after each entry.

## TITLE

*TOPAN packaging material destrap by INSTRON*

Sample ID	TOPAN
Test Type	Tensile
Number of specimens	15
Geometry	Rectangular
Test Date	2-Feb-06
Operator Name	Ilona
Units	Metric
Machine type	4200/4300/4400
Custom Test Label	
Data Rate (pts/sec)	6.667
Second Data Rate (pts/sec)	0
Crosshead Speed (mm/min)	10
Second Speed (mm/min)	0
Third Speed (mm/min)	0
Temperature (Deg F)	73
Humidity (%)	50
Sample comments	
Series IX version	8.31.01
Method	50

	Width (mm)	Thickness (mm)	Spec gauge len (mm)	Ext. gauge len (mm)
CONTROL	24.5	0.29218	110	190
CONTROL	24.5	0.29218	110	190
CONTROL	24.5	0.29218	110	190
PREHEATING	24.5	0.29218	110	190
PREHEATING	24.5	0.29218	110	190
PREHEATING	24.5	0.29218	110	190
HPP	24.5	0.29218	110	190
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HPP	24.5	0.29218	110	190
HPP	24.5	0.29218	110	190

Sign and date after each entry.

	Load at Peak (kgf)	Displiment at Peak (mm)	Displiment at Break (mm)	Displiment at 0.2% Yield (mm)	% Strain at 0.2% Yield (%)	Young's Modulus (kgf/mm <sup>2</sup> )	Energy at Yield (kgf-mm)	Energy at Break (kgf-mm)
Control	6.051	9.09	10.26	2.1102	1.91836	32.522	3.7626	48.5887
Control	5.458	9.84	19.2369	3.9117	3.55609	26.7686	8.09935	77.6443
Control	4.444	4.29	11.0664	3.408	3.09818	24.9133	5.9493	34.6389
Mean	5.317667	7.74	13.521	3.1433	2.85754	28.06797	5.937083	53.623966
HPP	7.774	14.47	15.47	3.1327	2.84791	34.2647	7.8521	95.2141
HPP	7.53	13.11	13.82	3.2136	2.92145	32.5495	8.31332	81.6517
HPP	7.232	11.79	13.1	1.9606	1.78236	34.4468	3.42282	74.2362
HPP	7.984	14.8	16.21	3.0396	2.76327	35.6731	7.75204	104.23
HPP	7.34	11.66	12.96	3.0137	2.73973	34.2243	5.31479	71.082
HPP	7.203	12.45	13	2.5626	2.32964	33.3021	4.76232	73.2985
Mean	7.509	12.97	14.0566	2.871967	2.61088	34.39983	5.94305	82.870166
Preheating	3.764	2.6	7.4	2.1886	1.98964	29.6361	3.57902	18.4103
Preheating	4.918	8.09	9.73	2.1603	1.96391	30.0843	2.9524	37.4879
Preheating	6.056	9.37	10.67	2.6066	2.36964	28.3979	4.84509	47.5081
Mean	4.912667	6.68666	9.26666	2.3185	2.10773	29.37277	3.79217	34.468766
Mean	6.31283	10.13	12.7436	2.77568	2.52335	31.3986	5.55043	63.6659
Standard Deviation	1.41105	3.7745	3.21823	0.606066	0.550969	3.40195	1.99927	26.0829
Mean - 2.00 Standard Deviation	3.49074	2.581	6.30714	1.56355	1.42141	24.5947	1.55189	11.5002
Mean + 2.00 Standard Deviation	9.13492	17.679	19.1801	3.98782	3.62529	38.2025	9.54897	115.832
Minimum	3.764	2.6	7.4	1.9606	1.78236	24.9133	2.9524	18.4103
Maximum	7.984	14.8	19.2369	3.9117	3.55609	35.6731	8.31332	104.23
Coefficient of Variation	22.352	37.2606	25.2537	21.8349	21.8349	10.8347	36.0201	40.9684
Median	6.6295	10.75	12.98	2.81015	2.55468	32.5358	5.07994	72.1903

Sign and date after each entry.

TITLE

MRE packaging testing by Istvan

Sample ID	MRE
Test Type	Tensile
Number of specimens	15
Geometry	Rectangular
Test Date	8-Feb-06
Operator Name	Ilona
Units	Metric
Machine type	4200/4300/4400
Custom Test Label	
Data Rate (pts/sec)	6.667
Second Data Rate (pts/sec)	0
Crosshead Speed (mm/min)	10
Second Speed (mm/min)	0
Third Speed (mm/min)	0
Temperature (Deg F)	73
Humidity (%)	50
Sample comments	
Series IX version	8.31.01
Method	50

	Width (mm)	Thickness (mm)	Spec gauge len (mm)	Ext. gauge len (mm)
CONTROL	24.5	0.29218	110	190
CONTROL	24.5	0.29218	110	190
CONTROL	24.5	0.29218	110	190
PREHEATING	24.5	0.29218	110	190
PREHEATING	24.5	0.29218	110	190
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HPP	24.5	0.29218	110	190
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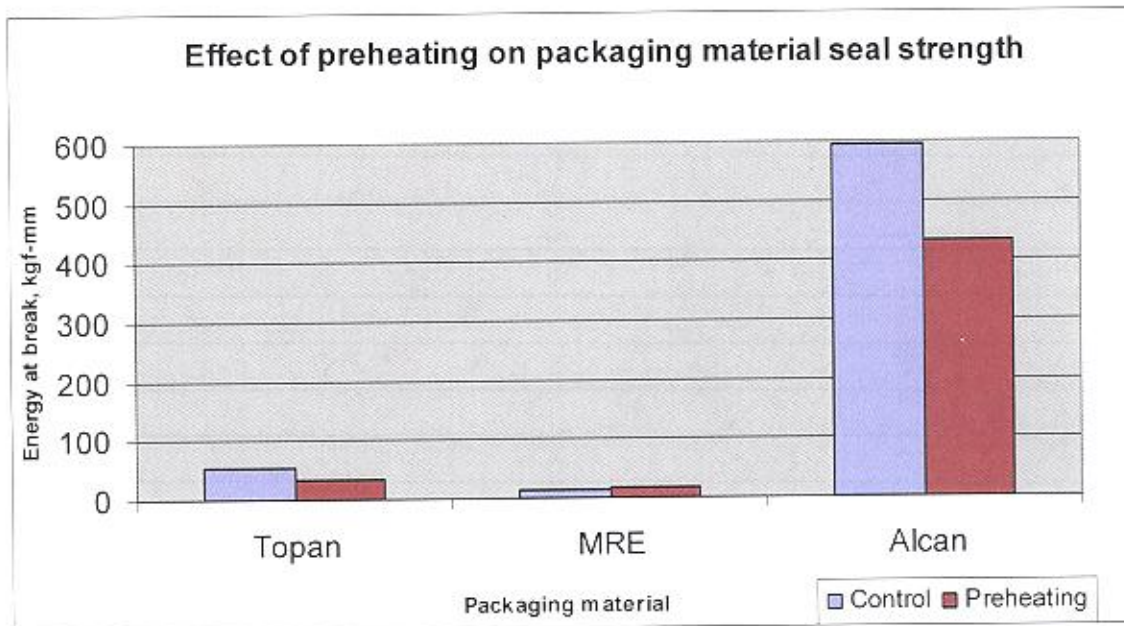
Sign and date after each entry.

	Load at Peak (kgf)	Displcmnt at Peak (mm)	Displcmnt at Break (mm)	Displcmnt at 0.2% Yield (mm)	% Strain at 0.2% Yield (%)	Young's Modulus (kgf/mm <sup>2</sup> )	Energy at Yield (kgf-mm)	Energy at Break (kgf-mm)
Control	3.254	3.91	4.36	3.439	3.12636	17.6697	4.54615	7.36876
Control	3.784	3.97	4.64	3.04	2.76364	20.0586	4.28068	9.77706
Control	4.569	4.64	6.6333	3.7333	3.39391	23.0956	6.41831	18.167
Mean	3.869	4.173333	5.2111	3.4041	3.094637	20.27463	5.081713	11.77094
HPP	4.264	6.46	8.0124	2.3373	2.12482	26.536	2.59498	23.7064
HPP	4.416	6.27	7.1829	1.8163	1.65118	28.641	1.95831	22.0984
HPP	2.89	3.27	4.2333	1.5832	1.43927	32.4449	1.55631	8.15085
HPP	0.9637	2.49	3.72	1.6606	1.50964	10.9687	0.287762	1.74092
HPP	1.336	2.55	4.33	2.3356	2.12327	10.7842	1.4977	3.12887
HPP	2.901	2.54	4.0167	1.6082	1.462	27.0321	1.70416	8.077
Mean	2.795117	3.93	5.249217	1.8902	1.718363	22.73448	1.59987	11.15040667
Preheating	5.495	4.02	6.158	3.8579	3.50718	26.0201	10.3309	20.0512
Preheating	5.052	4.69	8.7091	3.634	3.30364	25.3426	6.80278	25.4874
Preheating	3.769	3.89	5.4653	2.2653	2.05936	26.9433	3.01748	13.6927
Preheating	4.142	3.83	7.5624	2.3622	2.14745	23.1955	2.92489	20.4991
Preheating	4.566	4.86	6.0873	2.7873	2.53391	24.7871	5.28844	18.9865
Preheating	2.985	3.62	4.0603	2.5103	2.28209	17.9595	2.94291	7.14686
Mean	4.334833	4.151667	6.3404	2.902833	2.638938	24.04135	5.2179	17.64396
Mean	3.62578	4.06733	5.67807	2.59803	2.36185	22.7652	3.74345	13.8719
Standard Deviation	1.27029	1.19534	1.64182	0.788223	0.716566	6.20373	2.61178	7.83412
Mean - 2.00 Standard Deviation	1.0852	1.67666	2.39443	1.02159	0.928717	10.3578	-1.48012	-1.79628
Mean + 2.00 Standard Deviation	6.16636	6.45801	8.9617	4.17448	3.79498	35.1727	8.96702	29.5402
Minimum	0.9637	2.49	3.72	1.5832	1.43927	10.7842	0.287762	1.74092
Maximum	5.495	6.46	8.7091	3.8579	3.50718	32.4449	10.3309	25.4874
Coefficient of Variation	35.0349	29.3888	28.9151	30.3392	30.3392	27.2509	69.7694	56.4745
Median	3.784	3.91	5.4653	2.3622	2.14745	24.7871	2.94291	13.6927

Sign and date after each entry.

## TITLE

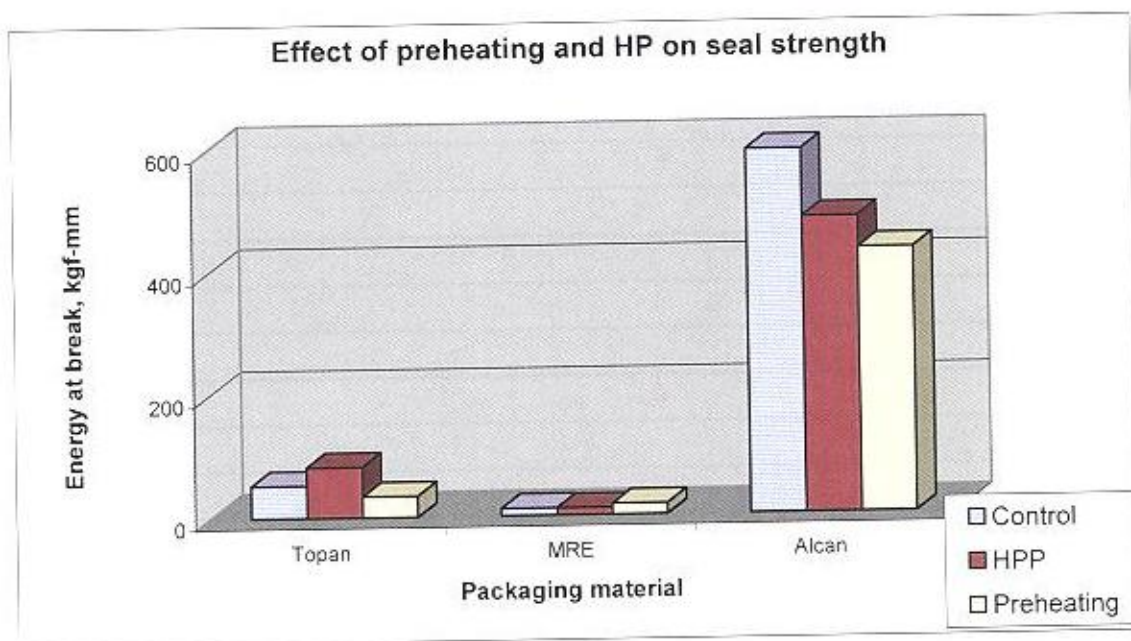
Comparison of preheating effect on packaging material seal strength



It was no significant effect of preheating on TOPAN AND MRE packaging materials while preheating for Alcan packages decreased energy needed to break seal.

TITLE

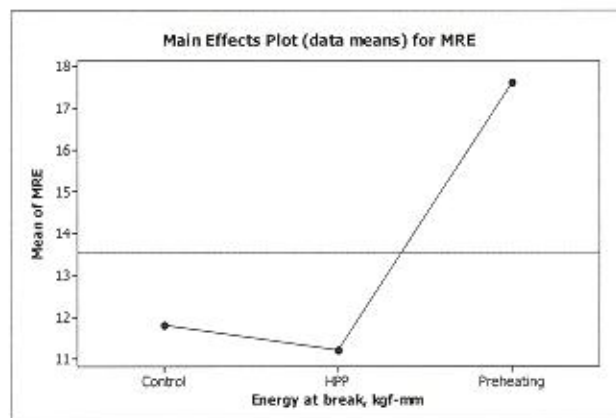
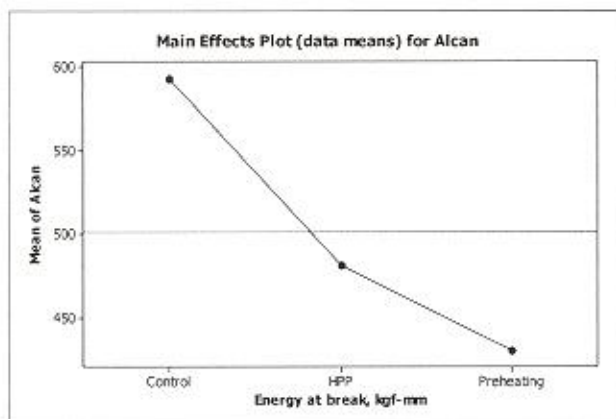
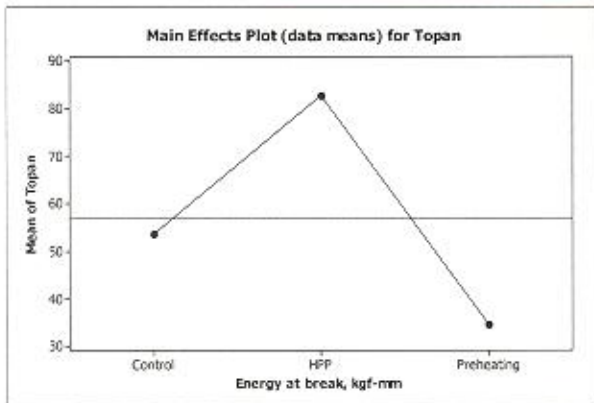
Comparison of preheating and High Pressure on seal strength of Topan, MRE and Alcan



It was no significant effect of preheating and high pressure on Topan and MRE. For Alcan preheating High Pressure decreased energy needed to break seal comparing to control sample while preheating decreased even more than high pressure. It shows that HPP effect is less than preheating.

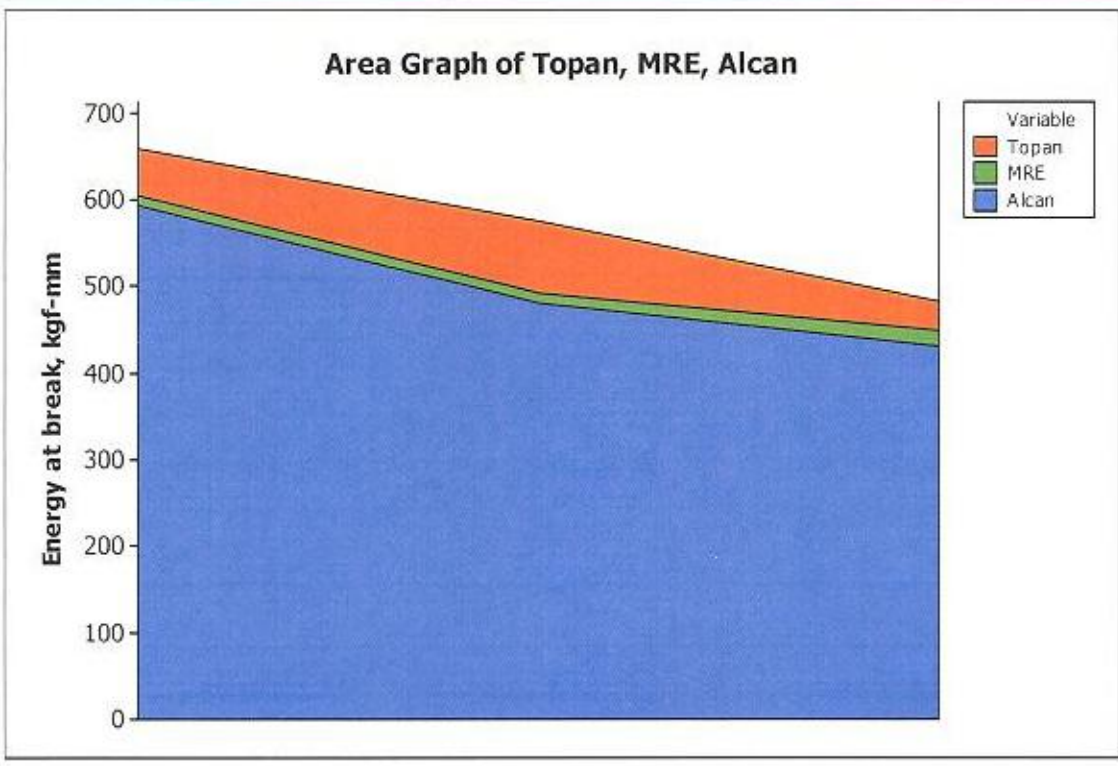


TITLE

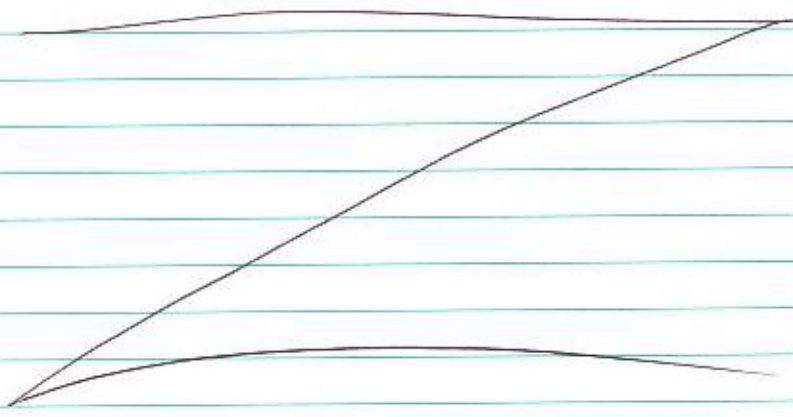


Sign and date after each entry.

TITLE



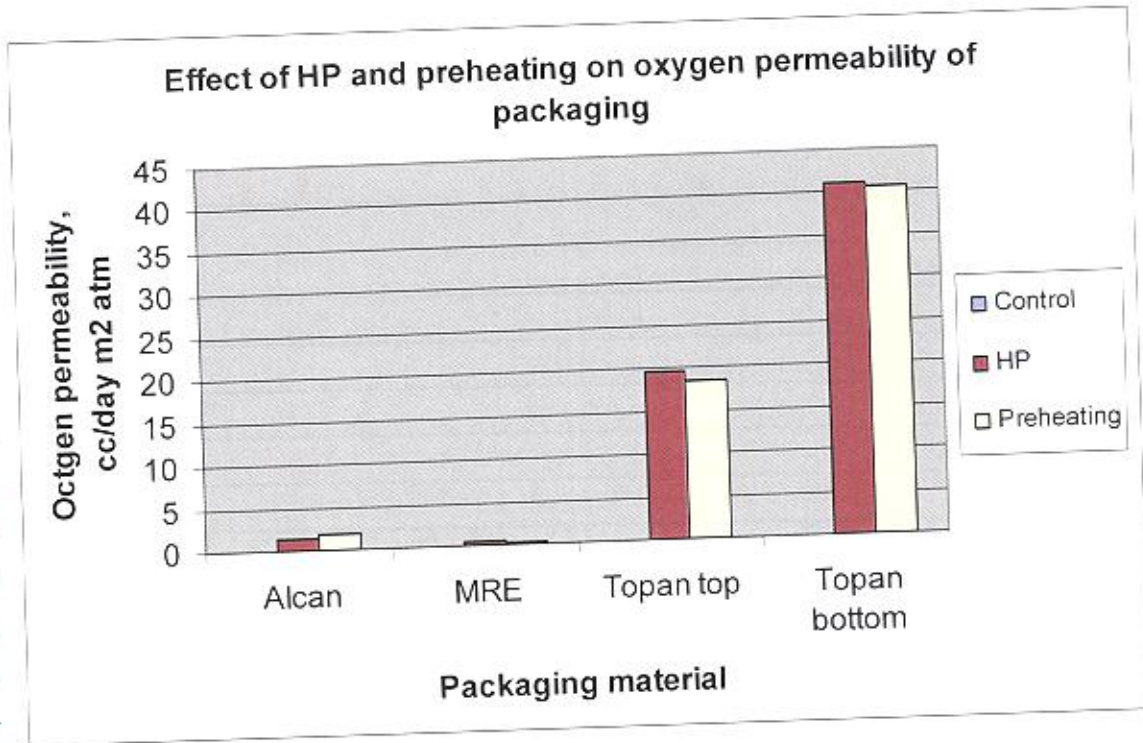
Comparing 3 tested materials: Topan, MRE, Alcan. Alcan showed the best fit for the project. Samples didn't blister, visually they looked better than Topan, which blistered during HP and MRE which didn't blister during HP but it was easier to break sep by INSTRON



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## TITLE

Permeability of oxygen by Mocon unit  
was tested for Alcan, MRE and  
Topan packaging materials



Since Topan top and bottom layers were different in structure, both sides were tested.

MRE showed lowest  $O_2$  permeability rate and it was no significant difference between control, preheated and high pressure treated samples.

Alcan packaging material also didn't show significant difference between control, preheated and HP samples. While Topan permeability changed a lot after preheating and HP treatment. Clear bottom of Topan packaging permeation rate was even higher with top.

TITLE

Oxygen permeation

	Control	HPP (cc/day m <sup>2</sup> atm)
Alcan	0.05/0.05/0.05	0.05/2.35/1.68
MRE	0.05/0.05/0.05	0.05/0.22/0.71
Topon top	0.05/0.05/0.05	0.05/19.2/19.7
Topon bottom	0.05/0.05/0.05	0.05/40.3/42.1

	Preheating
Alcan	2.04/2.0/1.58
MRE	0.31/0.2/0.5
Topon top	19/18.4/18.2
Topon bottom	39.9/41.2/40.4

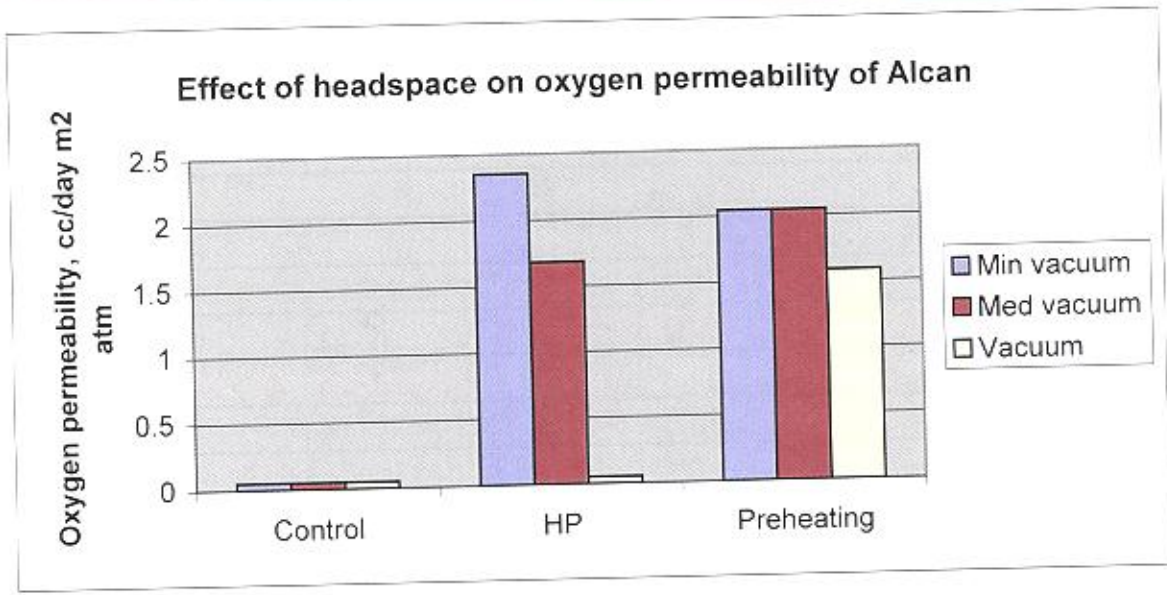
Since Alcan packaging was used to analyse different headspace effect during HP and preheating, O<sub>2</sub> permeability for packaging material with Minimum (Min) and Medium (Med) headspace was tested.

Alcan

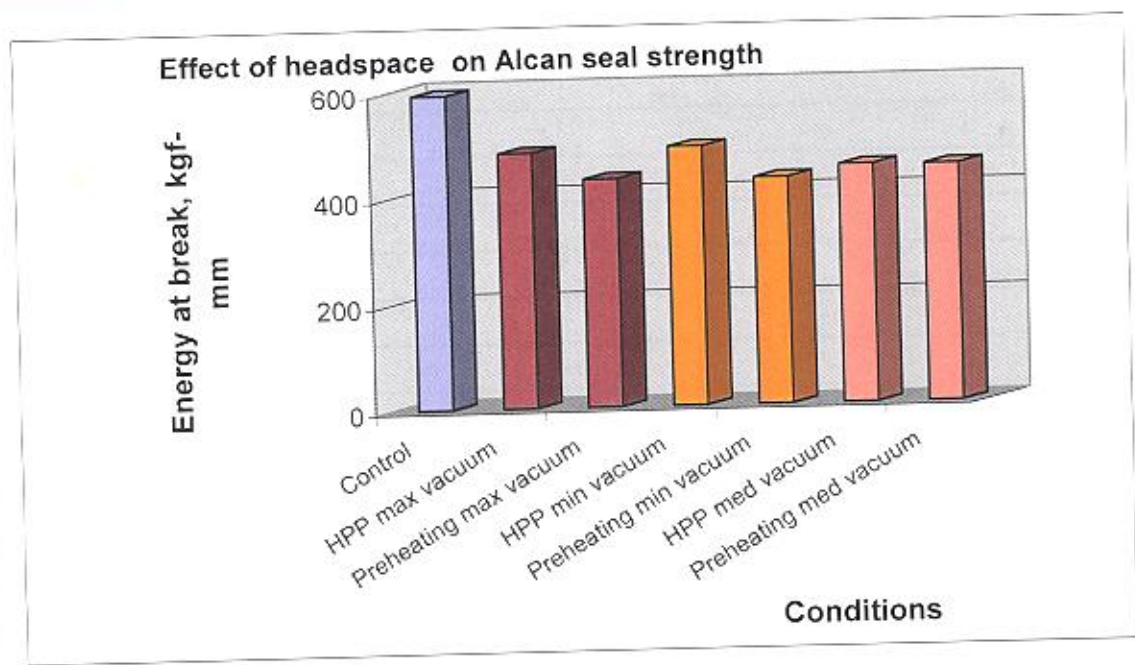
	Control	HP	Preheating
Min vacuum	0.05	2.35	2.04
Med vacuum	0.05	1.68	2.04
Vacuum	0.05	0.05	1.58

Packaging with Medium headspace didn't have such effect on oxygen permeability as comparing to Minimum headspace. But in both cases increase was not very significant.

TITLE



*Effect of headspace was tested for energy of break*



Oxygen penetration, was tested also by indophenol solution. Method is described below.

### Chemical Test of Oxygen Penetration

#### Summary

One of the fastest growing trends in the food packaging industry is the conversion from glass and metal packaging materials to ones made of plastic. There are many advantages in using plastic materials such as reductions in weight and cost, but the barrier properties of these material may be different from the conventional packaging systems, thus impacting the shelf-stability of the product.

A major concern for many products is oxidation. It is therefore important to know what packaging material is best for particular storage conditions, to know hot-spots for oxygen permeation and to understand the significance this will have on the food. The NCFST has developed a test to identify hot-spots for oxygen permeation in sealed packages. Selected pouches are filled with an redox indicator dye dissolved in agar. The pouches are then sealed to exclude air, and stored under the desired test condition. Oxygen permeation through the bag wall forces the dye to change from a clear, pale straw yellow color to blue. Bag regions high in oxygen permeation/gram of agar-dye turn blue fastest. The distribution of blue color clearly maps the course of oxygen ingress into the package.

#### Procedure

1. A 2,6 dichloroindophenole solution was made according to AOAC test 43.064 for ascorbic acid (AA).

The "indophenole solution" or IP is a blue oxidizing solution, which can oxidize approximately 0.016 mg per mL of IP solution. The concentration varies due to a insoluble fraction of the IP dye. The solution must be titrated against an ascorbic acid standard solution for exact measurement of IP concentration. However, for all but the most critical work this is not necessary since the endpoint is simply a qualitative color change

2. Approximately, 1 mg of AA is added to the IP solution per mL of solution.

Adding the AA reduces the IP solution causing the solution to change from blue to light yellow.

3. Agar (2%) is added to the IP solution which is taken to boiling to disperse the agar and then tempered to around 50°C in a water bath.

Sign and date after each entry.

## TITLE

4. The cooled ( $50^{\circ}\text{C}$ ) molten agar is poured into the container to be tested and immediately sealed to exclude air bubbles.

Excluding air bubble usually requires squeezing the package until all headspace gas is removed and then sealing through the IP agar solution.

### Principal

Oxygen migrating into the package oxidizes the AA. Once the AA is removed through oxidation (to become diketogulonic acid) the IP returns to its blue color. Each ascorbic acid requires 2 molecules of oxygen. Therefore, the amount of oxygen can be calculated from the amount of agar which has changed color.

### Sources of Error

Several forces work against exact quantization. Some AA is oxidized when agar is added with heating. Some small bubble entrapment is almost inevitable. These may remove some ascorbic acid causing the solution to change more quickly. There is always a little more oxygen migrated through package than indicated by the IP since all the AA in a region must be depleted before the color changes. Therefore, any oxygen acting on a source of AA which has not totally been removed does not result in a color change and consequently is not counted. Samples should be stored away from light since AA is susceptible to degradation by UV light.



### Adaptations of the Test

The IP test can also be used to compare the relative oxygen barrier of flat sheets. For this test a glass (not plastic) test tube is filled to a bulging meniscus with the tempered agar solution. A test patch of the polymer is placed over the mouth of the test tube and sealed air-tight using an cap which has an opening on top. The tube is inverted to allow any bubbles to rise to the butt end of the tube. Once the agar has solidified the tubes can be righted if desired. Color change is observed at the polymer/IP interface.

TITLE



### Test of Concept

Tubes were filled with IP solution as described immediately above. EVOH, PET, nylon, OPP and LLDPE were used to cover tubes. All films were nominally 1 mil thick except PET (1/2 mil) and OPP (0.92 mil). The ranking of oxygen permeation is well known for these structures and follows the pattern:

**EVOH < nylon < PET < OPP and < LLDPE**

LLDPE changed within several hours, OPP changed within 24 hours, PET required approximately 1 week. Neither nylon nor EVOH had changed within 2 weeks.



Sign and date after each entry.



## TITLE

Since Alcon and Topen were clear packages, this chemical, indophenol test could be used for these two type of packages.

In both cases it showed similar trends. Alcon and Topen showed no changes in 30 days, later after checking in 60 days, it was no changes detected.

## TITLE

## CALIBRATION TEST

OBJECTIVE :- To calibrate the K type thermocouple using three different temperature ( $0^{\circ}\text{C}$ ,  $100^{\circ}\text{C}$  &  $140^{\circ}\text{C}$ )

MATERIALS :-  
 Temperature bath of  $0^{\circ}\text{C}$  made through ice  
 Temperature bath of  $100^{\circ}\text{C}$  made through boiling water  
 Temperature bath of  $140^{\circ}\text{C}$  made through heated oil.

## METHOD :-

- A constant temperature was attained in all the three temperature bath (ice, water & oil)
- thermocouple was placed in the constant temperature medium with a ASTM certified thermometer. Thermometer and thermocouple should be at same level so as to get least variability in temperature.
- A attached data logger records the temperature at a regular interval
- A plot of thermocouple temperature curve shows its deviation from the actual thermometer reading.
- The plotted deviation should be corrected by making necessary adjustment.

## TITLE

## WEIDING OF THERMOCOUPLE

OBJECTIVE :- To weld K type thermocouple (chr-Al)

MATERIAL & METHODS :-

K type thermocouple (KT-TT-K-24-SLE) Chrome-Aluminium  
TL weld thermocouple & fine wire welder

JUNCTION - A smooth junction between the wires

LOCATION - NCFET

TEST DATE - JUNE