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CHLORATE-CANDLE FABRICATION BY HOT PRESSING

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CONTENTS

Abstract	ii
Problem Status	ii
Authorization	ii
INTRODUCTION	1
PHYSICAL FACTORS AFFECTING THE HOT-PRESSING TECHNIQUE	1
FACTORS AFFECTING THE CANDLE DENSITY	2
FACTORS AFFECTING THE STRENGTH OF CANDLES	3
INCORPORATING THE FIBER INTO THE CANDLE MIXTURE	5
HEATING BY CONDUCTION	5
HEATING BY RADIO-FREQUENCY INDUCTION	6
THERMAL CRACKING	9
A FEW CONSIDERATIONS IN CANDLE FABRICATION BY EXTRUSION	9
THE PERFORMANCE CHARACTERISTICS OF HOT-PRESSED CANDLES	9
CHEMICAL STUDIES OF NaClO_3 AND CANDLE COMPOSITIONS	11
Analysis of Active BaO_2 in Fabricated Candles	11
Analysis of the NaClO_3	12
Cause of Halogen Formation During Combustion	12
CONCLUSIONS	17
RECOMMENDATIONS	18
REFERENCES	18

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ABSTRACT

An investigation has shown that there are distinct advantages to manufacturing chlorate candles by a hot-pressing technique, omitting water as a binder. Small quantities of water trapped in a chlorate candle can result in the production of significant quantities of chlorine. By the hot-pressing method, high-density candles (2.5 g/cc) have been produced which yield the equivalent of 200 cu ft of oxygen (STP) from 490 cu in. of candle volume.

Two methods can be used for forming candles: molding and extrusion. Extrusion has not yet been attempted, since there are problems involving this method which have not been solved; however, this method may be the final choice for large-scale candle production. Molded candles are best formed at temperatures from 225° to 245°C at pressures from 10,000 to 5000 psi, respectively. A double-action ram can be used; with a single-action ram, successive pressing increments should be used to avoid uneven distribution of density. Use of a glass-wool blanket or oven annealing is recommended to avoid thermal stresses on cooling.

PROBLEM STATUS

This is a final report on one phase of the problem, work is continuing on other phases.

AUTHORIZATION

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CHLORATE-CANDLE FABRICATION BY HOT PRESSING

INTRODUCTION

Submarine chlorate oxygen candles as now made are pressed at room temperature using water as a solution-binding agent; this water is later removed by prolonged drying at elevated temperatures. A possible method that would avoid the use of water is to press the dry candle mixture at elevated temperatures. Candles produced by this technique are called hot-pressed candles. Other desirable features of the hot-pressing technique are higher candle density and improved resistance to both cracking and abrasion. This method is technically feasible because physical changes in the solid chlorate-candle mixture, such as softening, sintering, etc., occur before the melting point is reached (1).

Another possible candle-fabricating method is extrusion. If a heat-softened mass is continuously rotated, uniform distribution of the various ingredients will be maintained. This method should be applicable to mass production. However, a fire hazard may exist due to the inflammable nature of the candle mixture contained by the extrusion feeding assembly. Another major disadvantage that may be encountered is excessive frictional wear of the extruder's feeding screw by the fibrous ingredient.

This report will discuss physical factors affecting the hot-pressing technique, performance characteristics of hot-pressed candles, and chemical studies of NaClO_3 itself and NaClO_3 candle compositions. In order to facilitate the investigation of halogen production and the mechanism of sodium chlorate decomposition, a new volumetric procedure was devised and is described briefly in this report.

PHYSICAL FACTORS AFFECTING THE HOT-PRESSING TECHNIQUE

This section discusses physical properties that may influence candle fabrication, such as fiber type (glass or metal), fiber dimensions, effect of compaction pressure at various temperatures, etc. In order to investigate these factors a number of sample mixtures were made. All mixtures in this group contained 88 wt-% NaClO_3 , 4 wt-% iron powder, 4 wt-% barium dioxide, and 4 wt-% fiber; the batch size was 2500 grams. The deviations in the mixture were as follows:

- 1S Iron wool fiber, grade 00 (Brillo), average length 4 mm, average diameter 0.02 mm, weight-mean diameter (dw) 236μ (2)
- 2S Iron wool fiber, grade 2 (American Steel Wool Mfg. Co.), length 6mm, diameter 0.06 mm, dw 210μ
- 3S Iron wool fiber, grade 00-SF (American Steel Wool Mfg. Co.), length 0.7 mm, diameter 0.02 mm, dw 240μ
- 6G Glass fiber, length 0.7 mm, diameter 0.02 mm, dw 225μ
- 7G Differs from the 6G lot only in particle-size distribution; dw 400μ
- MSA Mixture 2S with 3% water added just prior to pressing (at room temperature). The cake was then dried at 115°C for 48 hours.

The weight-mean diameter (d_w) is the diameter of a particle whose mass multiplied by the number of particles in the sample equals the mass of the sample (2). To produce the metal fibers of various lengths, the strands were chopped in a blender. The fiber dimensions for each mixture are an average of 50 random measurements made with a microscope equipped with a filar micrometer eyepiece.

FACTORS AFFECTING THE CANDLE DENSITY

Figure 1 shows the effect of the pressing pressure on the density at various temperatures for mixtures 2S and MSA. All other mixtures show the same relationship.

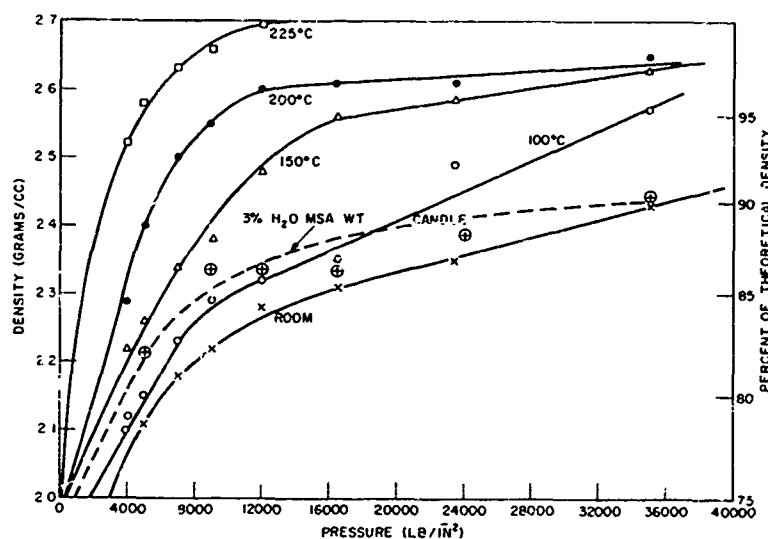


Fig. 1 - The effect of pressing pressure on density of candles, mixtures 2S and MSA

A pressing pressure of 4000 psi at room temperature compresses the powder only slightly, from 1.90 (uncompacted powder) to 2.06 g/cc. However, at 36,000 psi the density increases to 2.44 g/cc. At 10,000 psi and 100°C the density was 2.30 at 150°C 2.44, at 200°C 2.57, and at 225°C it was 2.68 g/cc. The theoretical crystal density, 2.70 g/cc, is reached at 13,000 psi and 225°C, while at 200°C this density is not obtained even at a pressure of 40,000 psi. A common density of 2.64 g/cc is reached at 40,000 psi for the temperature interval between 100° and 200°C.

From the above data it can be seen that the most practical pressing pressure in the 200° to 225°C range is 12,000 psi; doubling or tripling the pressure does not significantly increase the density. When the temperature is raised to 245°C the candle mixture becomes sticky, and only 4000 psi is required to produce 2.60 g/cc density (2S mixture); increasing the pressure to 7500 psi causes the cake to liquefy.

The diameter of the die used for obtaining the data in Fig. 1 was 2.36 cm. Cake height varied from 0.85 to 1.1 cm, and the weight of the cake was 10 grams. For pressing the cake a Carver laboratory press was employed; the pressure gauge was calibrated by an Ashcroft dead-weight gauge tester. The samples, plunger, and die were brought to the proper temperature in an oven and used immediately after removal from the oven.

Table 1 shows the effect of the height-to-diameter ratio on the density. The theoretical maximum density of the mixture used in this experiment is 2.69 (4% iron fiber, 4% iron powder, 4% BaO₂, 88% NaClO₃). The cake diameter was 2.85 cm; its weight, with an h/d ratio of 1.2, was 53 grams, and for h/d ratio of 2.4 the weight was 106 grams. A single-acting ram was used; i.e., the ram pressure was applied from one direction only.

Table 1
Effect of Height-to-Diameter Ratio on Density*

Approximate h/d Ratio	Temperature (°C)	Compaction Pressure (psi)	Density (g/cc)
1.2	200	5,000	2.34
1.2	225	5,000	2.40
1.1	225	10,000	2.66
1.1	200	20,000	2.68
1.1	225	20,000	2.68
2.4	200	10,000	2.38
2.4	225	10,000	2.40
		upper half of 7	2.60
		lower half of 7	2.20

*Mixture 4-4-4-88 (grade 00 iron wool); diameter of cake 1.125 inches; single-action ram; theoretical maximum density 2.69 g/cc; weight with 1.2 h/d ratio 53 grams, with 2.4 h/d ratio 106 grams.

Density results from the curves in Fig. 1 apply to an h/d ratio of about 0.4. For a ratio of 1.2, the cake density decreased an average of 0.12 g/cc at a pressing pressure of 5000 psi (200° to 225°C). When the h/d ratio was increased to 2.4 and the pressure correspondingly increased in the same temperature range, the density decrease remained nearly the same.

The use of a double-ram die (ram movement at both ends of the cake) on the large oxygen candle (h/d 1.84) should have approximately the same effect as a single-action press would on a cake at an h/d ratio of 1. In addition to use of a double-acting ram, raising the mixture temperature to 240° to 245°C should significantly lower the pressure required to produce a dense candle.

FACTORS AFFECTING THE STRENGTH OF CANDLES

Chlorate candles may be subjected to rough handling during transit and use; in addition, sufficient stress may be produced during burning to crack the candle and thus stop combustion. For these reasons, the cohesion of the individual particles is an important consideration, especially when water is not used as a bonding agent. Factors that may significantly influence the bond strength are: density, temperature during pressing, fiber length, particle-size distribution, and thermal stress on cooling.

The bond strength was measured by determining the force required to crush a ten-gram cake with diameter 2.36 cm and height varying with density from 0.85 to 1.1 cm.

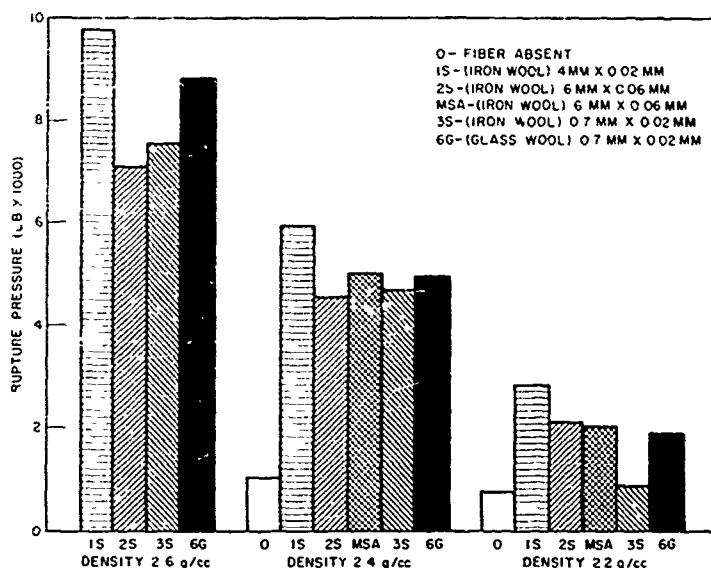


Fig. 2 - The effect of fiber and density on strength of chlorate candles

Figure 2 shows the effect of fiber and density on the strength of chlorate candles. The presence of fiber is necessary for holding the cake together, especially during combustion. A fiberless cake at low density would be expected to crumble readily, and increasing its density does not significantly improve the rupture strength, as shown by Fig. 2. The rupture strength of the 3S mixture at low densities is lower than the corresponding 6G (glass wool) mixture. This is probably related to the number of fiber particles; iron, having a higher density, would for the same fiber geometry yield a smaller number of particles per unit weight.

The relative rupture strength of wet-pressed candles should lie in the 2000 to 6000 psi range. The rupture pressures quoted here and in Fig. 2 are relevant only to the geometry of the ten-gram cake; different cake dimensions will change these values. Hot-pressed candles having a density of 2.5 g/cc or greater should have a relative rupture strength approximately twice that of wet-pressed candles and should be able to withstand considerable stress. This has been verified; hot-pressed candles (6-3/8 in. diameter, 2 in. high, weight 6 lb) have been dropped repeatedly from a height of 3 ft without cracking. Low-density candles (2.2 g/cc) could not tolerate this treatment.

When candles are pressed at temperatures lower than 225°C, the particle-size distribution plays a significant role in the particle cohesion (Fig. 3). Samples 6G and 7G differ only in their particle-size distribution; 35% is retained by a 50-mesh screen for mixture 6G, and 54% for mixture 7G. In both cases 100% passes a 20-mesh screen (U.S. Standard Sieve Series).

The relative bonding strength of the 7G candles is only a quarter of that of 6G candles, even at a density of 2.4 g/cc. Decreasing the particle size significantly below that in the 6G mixture did not appreciably increase the strength. Therefore, at pressing temperatures below 225°C, a wide distribution of particle sizes below 50 mesh is essential for producing a shock-resistant candle. The particle-size distribution does not affect the candle strength at temperatures above 225°C, since fusion occurs.

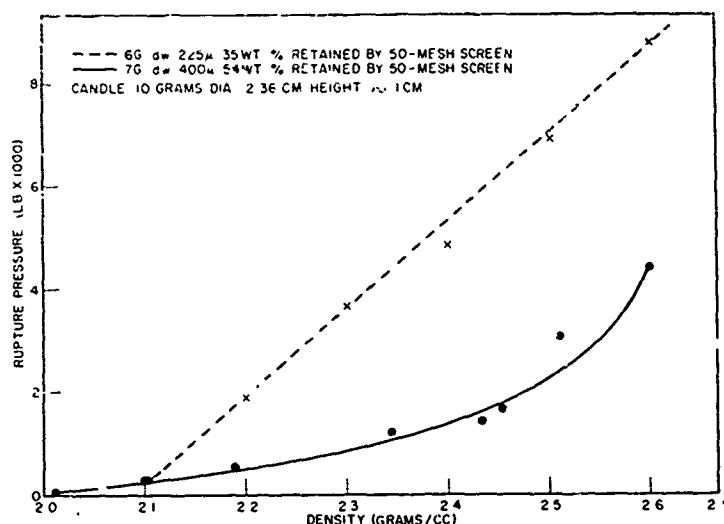


Fig. 3 - The effect of particle size on bond strength

INCORPORATING THE FIBER INTO THE CANDLE MIXTURE

Metal fiber was difficult to mix into the powder, since agglomeration occurred if the fiber length was 1 mm or more. Pouring the candle mixture from one vessel to another also caused the fiber to separate, with the exception of mixture 3S, which could stand pouring but not shaking. It was found that a ball mill would distribute the fiber, provided that a volume of glass beads (4 to 5 mm) equal to the volume of the candle mixture was used. The glass beads were later removed by careful screening. This procedure, however, was time consuming and did not altogether eliminate the agglomeration of the fiber.

Later, ball milling was discarded and mixing was accomplished by shaking the metal fiber, 00 grade, NaClO_3 powder, and other candle ingredients simultaneously through a 10-mesh screen. This eliminated agglomeration, but the fiber tended to settle in layers. This was prevented by attaching a 6-mesh screen under the 10 mesh.

HEATING BY CONDUCTION

A thermal-conductivity coefficient (k) for crystalline NaClO_3 of 2.665×10^{-3} cal/sec (cm^2) ($^\circ\text{C}/\text{cm}$) is given in Lange's Handbook of Chemistry (3). Evcken (4) gives a value of 2.665×10^{-3} cal/sec (cm^2) ($^\circ\text{C}/\text{cm}$) at 0°C and a value of 3.77×10^{-3} at -78°C . The value of the thermal-conductivity coefficient at operating temperatures and densities would be useful in designing heating equipment for hot-pressing or extrusion. Therefore, a crude measurement was undertaken by two different methods. One method, which gave rather questionable results, was to pass heat radially inward from an electric heater, through sodium chlorate to a flowing-water heat sink. Corrections were large, and the data were discarded in favor of those obtained by a comparison method using the conductivity of stainless steel (Fig. 4). In this method iron-constantan thermocouples were used. A variac-controlled hot plate was the heat source. The ends of stainless steel and copper cylinders were polished to a mirror surface; aluminum foil was used to ensure good contact between the steel and sodium chlorate. A two-inch-thick glass-wool blanket surrounded the apparatus to reduce heat loss.

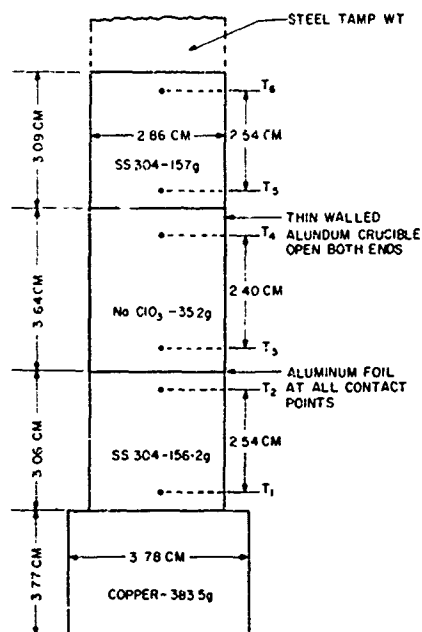


Fig. 4 - Thermal-conductivity-apparatus comparison method

Calculations of the thermal-conductivity coefficient of NaClO_3 using the comparison-method data gave $3.52 \times 10^{-3} \text{ cal/}(\text{sec})(\text{cm}^2)(^\circ\text{C}/\text{cm})$ at a density of 1.7 g/cc (apparent density of NaClO_3 powder) and in the temperature range of 130° to 210°C .

Since the heat-transfer rate of sodium chlorate is low, long heating periods may be involved. The effect of long-duration high temperatures on candle compositions was studied. A hot-pressed 2550-gram candle (composition by weight 88% NaClO_3 , 4% glass fiber, 4% iron powder, and 4% BaO_2) was placed in a 235°C oven for eight days. The loss in weight was 8.4 grams (0.33%). Chemical analysis indicated that a major fraction of this loss (6.5 grams) was oxygen from BaO_2 . Complete removal of water in high-density candles is very difficult, and traces of water in the NaClO_3 probably affect the decomposition of BaO_2 when held at high drying temperatures (235°C) for long periods.



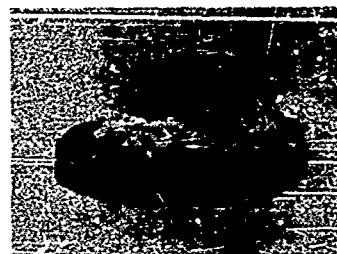
Less than 0.3% H_2O in the chlorate could liberate 6.5 grams of O_2 . Sodium chlorate, BaO_2 , or mixtures of BaO_2 and NaClO_3 (not pressed into a dense cake) do not decompose significantly at this temperature. Therefore, practical periods of heating of unpressed candle mixtures at temperatures up to 235°C are feasible as far as stability is concerned. Mixture temperatures, even when dry, should not exceed 240°C for long periods, because accelerated crystal growth starts at this point (Fig. 5).

Due to the low heat-transfer rate and crystal growth, static batch heating by conduction does not appear to be feasible. However, heating by conduction, even in the 240° to 250°C range, is feasible provided that a dynamic method such as extrusion or a rotating drum is used.

HEATING BY RADIO-FREQUENCY INDUCTION

Radio-frequency heating offers a solution to the problem of batch heating of candle mixtures. Suitable pressing temperatures can be reached in a matter of minutes. Candle

Fig. 5 - Crystal growth of a candle mixture at 250°C (2-1/2 hr)



mixtures were heated by a 10-kw Westinghouse radio frequency generator operating at 450 kilocycles. Various container diameters from 1-1/4 to 6 inches were employed. However, the best system geometry with regard to power consumption, uniformity of heating, convenience, and batch volume was as follows. Dimensions of the induction coil (copper tubing, 3/8-in. diameter, water cooled) are 7-3/4 in. in diameter by 5 in. high, with 0.7 turn per inch, where the container is a 4000-ml Pyrex beaker 6 in. in diameter, holding 5.5 lb of candle mixture packed around a centrally positioned 3-in.-diameter steel tube (SS304, 1/16-in. wall thickness) to a depth of 4-3/4 in. The hollow center tube aids in reduction of hot spots because it equalizes temperatures by conduction. Thermocouples were employed for temperature control. Another important factor in the elimination of hot spots is the uniformity of distribution of metal fiber.

As one would expect, it was found that the mixture composition strongly influences the coupling efficiency. A mixture employing 4% iron as powder would not couple to any significant extent; however, Fig. 6 shows a 2500-gram hot-pressed candle containing 4% iron as fiber which was heated entirely by rf induction.



Fig. 6 - Hot-pressed induction-heated candle. A round 6-inch-diameter candle was split lengthwise through the center.

Induction heating does not appear to be feasible for in-place casting of candles (heating the mixture to its melting point in the mold). At temperatures high enough for casting (255°C and above), slow decomposition occurs. Examination of the interiors of candles made by this method showed numerous gas pockets, resulting in a low density (about 1.70 g/cc). Variations such as changing the brand of iron wool, changing the source of BaO₂, and changing the induction-heating rate did not help. The bubbles appear to arise at the

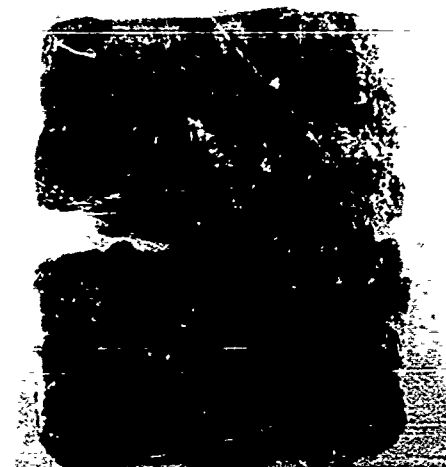


Fig. 7 - The suppression of bubble formation on in-place induction-cast candles by 0.5 percent KClO_4 . The lower sample contains 0.5 percent KClO_4 ; the dark spots in this candle are clumps of iron wool. The upper sample, to which perchlorate was not added, shows definite bubble formation.

liquid-solid NaClO_3 interface; bubbling decreases almost to zero when the NaClO_3 solid phase disappears.

Addition of potassium perchlorate does reduce the bubble formation. (Note on Fig. 7 that the dark spots of the KClO_4 -treated candle are not air pockets but are bits of agglomerated metal fiber.) However, this effect occurs only at temperatures below 255°C . The eutectic for KClO_4 - NaClO_3 compositions seems to be near 10% KClO_4 ; the melting point is with this mixture about 233°C . The optimum KClO_4 concentration as far as burning, economics, and efficiency are concerned is 0.5 wt-%. In addition to decreased bubble formation, other advantages of KClO_4 formulations are: a more even burning rate, elimination of chlorine traces in the evolved oxygen, greater candle strength, and increased resistance to thermal cracking.

Small-diameter candles (3 in. or less) made by pouring the molten mixture into molds may advantageously incorporate induction heating. Figure 8 shows a candle cast by pouring the induction-heated melt with agitation into an aluminum pipe of 3 in. I.D. The 1300-gram cake shrank on cooling and was easily removed from the pipe. Bubble formation did not occur; a possible explanation for this fact may lie in the rapid cooling that took place. On burning a 600-gram section of this candle no trace of chlorine could be detected by a $1/4$ Drager chlorine "sniffer tube." The density of this candle was 2.30 g/cc.

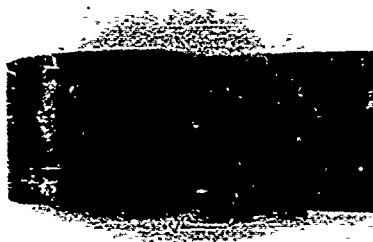


Fig. 8 - Two halves of an induction-heated candle cast by pouring into a mold

THERMAL CRACKING

Hot-pressed candles, after removal from the die, were subject to thermal stress on cooling. In most cases a thin crack developed within ten minutes. Cracks extending the length of the cake started at the surface and on further cooling progressed inward to the center. Generally, the process stopped at this point. This problem was solved by wrapping the candle with a glass wool blanket, or otherwise reducing the initial cooling rate. With 5-1/2-lb candles, this insulation period could be as short as 20 minutes; large candles will probably require more time.

A FEW CONSIDERATIONS IN CANDLE FABRICATION BY EXTRUSION

Many of the factors considered in the hot-pressing technique apply also to extrusion processing. For example, the iron-fiber length should be short (about 1 mm) to avoid settling. In addition, KClO_4 (0.5%) would be an aid as an anti-agglomerating agent because it brings about a partial melting at a lower temperature. This in turn causes the steel fiber to adhere to the particles and thereby reduces the tendency of the fiber to settle out.

The extrusion system must take into account thermal stress. With proper design this problem can be solved. In doing so, the time period between actual mixture extrusion and cutting the cake to the proper length should be as short as possible.

THE PERFORMANCE CHARACTERISTICS OF HOT-PRESSED CANDLES

Candle compositions were heated and pressed in a steel die. The dimensions were: height 19 cm, I.D. 16 cm, O.D. 22.6 cm. Two 35-ft lengths of insulated heating wire connected in parallel were wrapped around the die; at 110 volts the power input was 1050 watts. Asbestos-paper thermal insulation next surrounded the lateral surface, and the final protective layer was glass tape.

Candle mixtures in 2500-gram batches were pressed at ram pressures of up to 75 tons for one minute at temperatures ranging from 200° to 245°; generally the pressing temperature was at 225° C. The dimensional range of the resultant cakes was: height 4.7 to 5.1 cm and volume 960 to 1040 cc.

Candles were burned in a cylindrical stainless steel container with a welded bottom and bolted lid. A fiberglass filter was mounted on the outlet for NaCl smoke removal. The oxygen yield was measured by three methods: loss in weight of the complete burner unit, loss in weight of the candle itself (corrected for salt loss), and by a rotameter during O_2 evolution. The gain in weight of the salt-filter assembly gave a measure of the smoke produced. Chlorine and carbon monoxide were evaluated by Dräger "sniffer tubes."

Various candle compositions were investigated; the average oxygen yields of the following two mixtures: (a) 4% metal binder, 4% BaO_2 , 4% iron powder, and 88% NaClO_3 , and (b) 4% steel wool, 4% BaO_2 , and 92% NaClO_3 (a recent MSA Research Corporation formulation omitting iron powder) were 39.1 wt-% and 40.8 wt-% respectively. The average values for the impurities in the evolved oxygen were: chlorine less than 0.2 ppm when one type of metal fiber (grade 2 steel wool) was used and 0.2 ppm for another type (grade 00 steel wool), CO was 5 ppm and salt on the filter totaled 6 grams. When glass fiber was substituted for metal binder, the Cl_2 content rose to 25 ppm.

The peak gas evolution rate, for the 4-4-92 mixture, reaches about 4 cfm, and the integrated average for the entire candle is 2.5 cfm; with 1/2% KClO_4 (mixture 4-4-91.5) the values were maximum rate 3.5 cfm and average 2.0 cfm. For the 4-4-4-88 composition, the average maximum rate was 4.3 cfm, and the integrated average 2.4 cfm; when another type of metal fiber (grade 00 steel wool) was substituted, the values were 5.2 and 2.7 cfm.

The most efficient candle geometry on the basis of storage space would be a square or rectangular candle. Table 2 shows the candle weight and oxygen yield given by a rectangular candle $6\text{-}9/16 \times 6\text{-}9/16 \times 11\text{-}3/8$ in. (488 cu in.) at various densities. Limits of this size produced by MSA gave approximately 150 ft of O_2 , weighed 33 plus pounds, and had a density of 1.9 g/cc. The ratio between a square and a circular cross section is 0.786. The results for 5.5-lb cylindrical candles converted to square-candle geometry (488 cu in.) are shown in Tables 2 and 3. A 4-4-0-92 candle with a density of 2.43 g/cc would weigh approximately 43 lb (Table 2) and yield 199 cu ft of oxygen. The maximum burning rate should be around 6 cfm, and the integrated rate about half of that. The burning period should average 65 minutes and salt-smoke evolution about 50 grams. The 1/2% $KClO_4$ candle should have a maximum rate around 5 cfm, and an average rate of 2.5 cfm.

Table 2
Oxygen Yield versus Candle Density*

Density (g/cc)	Candle wt(lb)	O_2 yield, STP (cu ft)
1.9	33.6	151
2.0	35.4	159
2.1	37.3	167
2.2	38.9	175
2.3	40.7	183
2.4	42.5	191
2.5	44.3	199
2.6	46.1	207

*Square candle 490 in³; mixture 4% BaO_2 , 4% (metal fiber) 92% $NaClO_3$; weight percent oxygen evolved 40%. The candles were 5.5-lb cylinders; the results shown here were converted to square-candle geometry equivalent to $6\text{-}9/16 \times 6\text{-}9/16 \times 11\text{-}3/8$ in. (488 cu in.).

Ignition systems were not studied. However, it was noticed that a small quantity of sodium chloride spread over the ignition surface of the candle (i.e., 10 grams over 204 sq cm) aided ignition. Salt retards the flow of molten sodium chlorate and may also furnish a catalytic surface. The incorporation of a thin NaCl layer between the sodium chlorate and the igniter could significantly improve the ignitability of cold candles.

Investigations of catalytic mixtures and attempts to develop a fiberglass candle elucidated the roles played by iron fiber and BaO_2 . The functions that a metal fiber performs, other than furnishing some heat of combustion, are: (a) response to radio-frequency radiation, (b) transmission of heat into candle layers immediately beneath the combustion zone, thus propagating the reaction, and (c) binding of the candle at the burning front to prevent cracking. Glass-fiber candles and especially fiberless candles containing even 5% iron powder would not burn well. Metal fiber has many of the characteristics of an ideal bonding agent, such as low water content, a factor in Cl_2 production, and high melting point. Barium dioxide's role other than holding back Cl_2 is that of a partial substitute for iron, probably through a catalytic effect on one or more of the reactions involved. With BaO_2 absent or reduced, additional percentages of iron were required.

Table 3
Oxygen Production from Hot-Pressed Candles*

Composition†	Oxygen (wt-%)	Density (g/cc)	O ₂ Yield, STP‡ (cu ft)
(2) 4-4-4-88	38.6	2.49	191
(2) 2.5-4-4-89.5	39.8	2.56	205
(2) 4-4-4-88	38.8	2.61	200
(2) 4-4-4-88	38.9	2.57	198
(00) 2-4-4-90	39.8	2.51	201
(00) 4-4-4-88	40.0	2.55	203
(Glass) 4-4-4-88§	39.4	2.40	187
(00) 4-4-0-92§	40.6	2.46	199
(00) 4-4-0-92§	40.0	2.40	196
(00) 4-4-0-92§	41.0	2.42	199
(00) 4-4-0-92§	41.0	2.48	202
(00) 4-4-0-92§	41.0	2.43	199
(00) 4-4-1/2KClO ₄ -91.5	41.0	2.43	199

*Cake 5.5 lb, 6-3/8 in. diameter, pressed at 210°-225°C and 4700 psi.

†Composition: type of binder, percent binder, percent BaO₂, percent iron powder, percent NaClO₃.

‡Computed from results of 5.5-lb samples of same efficiency and density.

§New formulation of MSA Navy candles.

CHEMICAL STUDIES OF NaClO₃ AND CANDLE COMPOSITIONS

Analysis of Active BaO₂ in Fabricated Candles

The determination of the active BaO₂ (in reagent BaO₂) by itself can be accomplished by suitable methods (5a) without difficulty. However, when BaO₂ is incorporated into candle compositions, standard methods cannot be employed because: (a) the metal ingredient interferes with the BaO₂ determination in acid media, (b) separation methods employing neutral or alkaline solutions only partially dissolve the BaO₂, and (c) if solubilization were affected, the large amount of sodium chlorate present would seriously interfere with the BaO₂ determination.

The following method was found to give an estimate of the active BaO₂ present. Two 2-gram samples (pulverized) from the oven-heated candle mentioned in the section titled "Heating by Conduction" (235°C, 8 days) were added to Erlenmeyer flasks containing 25 ml 0.1 N As₂O₃, 75 ml of distilled water, and 3 grams of NaHCO₃. The solution was boiled for 30 minutes and allowed to stand overnight, and then filtered. The filtrate was back titrated with 0.1 N I₂ solution.

Sample 1 - gave 5.8 mg of active BaO₂ (0.29%)

Sample 2 - gave 6.2 mg of active BaO₂ (0.31%)

Control - gave 64 mg of a possible 72 mg of BaO₂

These results (samples 1 and 2) compare favorably with the calculated value derived from the loss in weight of the candle after correcting for NaClO_3 decomposition. The decomposition rate of NaClO_3 at 220° to 230°C was approximately 0.01% per day (experimentally determined by oxygen loss). The 2550-gram candle contained 2250 grams of NaClO_3 , therefore $2250 \text{ grams} \times 0.01\% \times 8 \text{ days} = \text{loss of } 1.80 \text{ grams } \text{O}_2 \text{ from } \text{NaClO}_3 \text{ decomposition}$. The total loss in weight was 8.4 grams; $8.4 - 1.8 = 6.6$ grams loss in weight from other sources. If this loss in weight was the result of BaO_2 decomposition ($\text{BaO}_2 + \text{H}_2\text{O} \rightarrow \text{Ba(OH)}_2 + 1/2 \text{ O}_2$), this would correspond to 69.8 grams of decomposed BaO_2 . The active BaO_2 content of the reagent used in making the candle was assayed (by an iodometric procedure) at 75 percent; for the 102 grams of reagent incorporated into the mixture, this would amount to 76.5 grams of actual BaO_2 . Therefore, $76.5 - 69.8 = 6.7$ grams of BaO_2 remaining, or 0.26 percent as compared with 0.30 percent by the analytical method.

Analysis of the NaClO_3

Candle compositions containing glass wool or asbestos as binders evolved potentially dangerous concentrations (1 ppm and higher) of chlorine (or chlorine oxides, such as Cl_2O , ClO_2 , ClO_3 , or Cl_2O_7 , none of which was specifically detected). The development of a rapid analytical method for the sequential analysis of halogen compounds (Cl^- , OCl^- , ClO_2^- , ClO_3^- , BrO_3^-) in sodium chlorate was undertaken in order to elucidate the mechanism responsible for halogen formation. Such a method, with modification, could have possible application in the rocket-propellant field for the assay of oxyhalide impurities in NH_4ClO_4 and LiClO_4 oxidizers (6).

Analytical methods exist for the quantitative determination of individual halogen components, such as Cl_2 , OCl^- , ClO_2^- , ClO_3^- , etc., and various combinations of them (5b, 7). However, a necessary requirement of the analytical scheme employed is the detection of extremely small quantities which, in turn, requires a large sample. A method was devised which allowed all analyses to be performed on a single sample.

The sodium chlorate used in these candle fabrications was rigidly analysed by this sequential combination of standard chemical techniques for Cl^- , OCl^- , ClO_2^- , ClO_3^- , and BrO_3^- . The results, shown in Table 4, agree quite well with the manufacturer's analyses and indicate that the halogen contamination in the evolved oxygen is not from impurities.

Cause of Halogen Formation During Combustion

In order to ascertain whether or not impurities in agricultural-grade NaClO_3 were responsible for halogen formation, a detailed analysis (volumetric and spectrographic) was performed. The results are summarized in Table 4.

As mentioned previously and summarized in Table 4, the amount of contaminants in the sodium chlorate could not account for the impurities found in candle oxygen. The halogen must arise, therefore, from the decomposition of sodium chlorate through a side reaction. The most probable halogen-producing intermediates would be sodium hypochlorite (8,9) and sodium chlorite (10). An experiment was devised to determine this point. Sodium chlorate alone, or with various admixed candle ingredients, was placed in a porcelain crucible with a thermocouple junction embedded in it. Ignition was initiated by heating from the bottom with a gas burner. Reaction was allowed to proceed for about 30 seconds, at which time it was quenched. The crucible contents were then pulverized and analysed for chlorite and hypochlorite. Assuming maximum liberation of chlorine from these contaminants, the resultant concentration of chlorine in the oxygen evolved from the sodium chlorate was calculated.

Table 4
Detailed Analysis of Chlorate Candle Ingredients,
Agricultural Grade NaClO_3 , 99.14% Pure

Sodium Chlorate		BaO_2		Steel Wool
Cations	Anions			
Mg (Residue analyzed found MgO) 0.6%	% Cl 0.08%	BaO_2 (active) 74.43%	74.43%	Grease 0.1%
Si 0.001 $\geq$ 0.004%	% $\text{BrO}_3 < 0.01$	BaO or Ba(OH)_2 [as Ba(OH)_2]	18.07%	
Al * $< 0.001\%$	ClO_2 absent	Cl^- (listed)	0.03%	C (alloyed) $< 0.1\%$
Ca $< 0.01\%$	OCl absent	(N cpds) (listed)	0.01%	Mn 0.1 - 1.0%
Fe* $< 0.01\%$		Ca^+ alkalies (listed)	0.50%	(manganese)
K $< 0.01\%$		Metal cations	0.01%	
Cr* $< 0.001\%$		Other as sorbed H_2O	6.95%	
		Total	100.00%	

NOTE - Water about 0.1 to 0.3%.

*Potential halogen producers if present as metal chlorate.

Table 5 shows that iron, NaCl, or combinations of iron-NaCl, $\text{NaCl-H}_2\text{O}$, NaCl-NaClO_3 , and iron- NaClO_3 (4 percent or lower) are not responsible for intermediate compound formation. However, factors such as the physical state of the candle (5A powdered and 5A pressed) and the water content (1D and 2D) appear to be significant.

Table 6 shows that only water that is prevented from rapid evaporation from the candle interior during combustion is capable of producing large amounts of NaOCl , NaClO_2 , or both. Samples 1F, 2F, 3F, and 4F all yield about the same available chlorine irrespective of the water content; with ignition occurring on the top surface of the candle mixture, water in these cases (1F-4F) was able to evaporate quickly. In 5F and 6F, the top (external) surface was coated with a layer of fused NaClO_3 and NaCl respectively; the candle mixture was underneath the sealed layer, and rapid water evaporation was thus restricted. The available chlorine rose to 160 ppm in the NaClO_3 sealed layer, and to 325 ppm under the NaCl seal. (The term available chlorine refers again to chlorine that would be formed if the oxyhalites present reacted to the maximum extent with water, NaCl, or both to produce chlorine.)

On the basis of melting points of these "sealants" one would expect the fused NaCl to be more effective and therefore to hold back a larger available chlorine content. Table 6 shows this concept to be valid. When the water content was lowered to a trace amount, as in sample 7F (top surface sealed with NaCl and ignited in the same manner as 5F, 6F samples), the available chlorine dropped to 40 ppm.

Tables 5 and 6 give the predicted Cl_2 values based on the analytically determined concentrations of NaOCl and NaClO_2 ; Table 7 shows the Cl_2 gas actually evolved from decomposition of NaClO_3 under conditions similar to the samples in those of Tables 5 and 6. The evolved chlorine corresponds fairly well with the available chlorine content of the hypochlorite and chlorite salts. This chlorine gas was measured by 1/a Dräger chlorine detector tubes as follows: The evolved oxygen from a sample of chlorate contained in a pyrolysis tube (or steel crucible) was aspirated into a 1000-ml Erlenmeyer flask and mixed with chlorine-free air. The gas volume was then large enough for an

Table 5
Effect of Candle Ingredients on Available Chlorine

Sample	Remarks*	Reaction Temperature (°C)	NaClO (wt-%)	NaClO (wt-%) ²	Available Cl ₂ † at Reaction Zone (ppm by vol)
4A	Powdered 4% Fe	790	0	0	0
5A	Pressed 4% Fe	780	0	0.018	80
6A	Powdered 7% Fe	760	0	0.018	80
7A	Powdered 4% Fe, 3% Al	780	0.011	0.006	85
8A	Powdered 12% Fe	780	0	0.020	85
9A	Powdered 20% Fe	780	0	0.018	80
1B	Cast 12% Fe	810	0	0.014	60
1C	Dry	830	0	0	0
2C	2% H ₂ O	810	0	0	0
3C	14% Fe	810	0	0	0
4C	14% Fe, 2% H ₂ O	840	0	0	0
1D	Dry	820	0	0.005	20
2D	0.3% H ₂ O	820	0.015	0	80

A Powdered candle mix 88% NaClO₃, 4% Fe, 4% glass wool, 4% BaO, 50 grams.

B NRL cast candle, 78% NaClO₃, 12% Fe, 6% glass fiber, 4% BaO₂, 50 grams.

C NaCl; O₂ introduced into molten mass 850 cc/minute, 24 grams.

D Reagent NaClO₃, 44 grams.

* Combustion interval 30 seconds, sample immediately quenched.

† Content of oxyhalites (NaClO and NaClO₂) and available chlorine based on formation at the reaction zone. The reaction zone is considered to occupy 20% by volume of the candle (ignition site plus the heat layer underneath comprises the reaction zone).

accurate division of the gas stream as follows: 1/10 passed through a Drager tube and then through a flowmeter, and 9/10 through another (dummy) Drager tube and flowmeter. The flow was continued until the chlorine indicated by the detector remained unchanged. The chlorine concentration in parts per million could then be calculated by computing the volume of chlorate oxygen that passed through the sampling tube.

Halogen formation apparently results from sealed H₂O-NaClO₃ reactions, the water having been retained above a threshold temperature. This would occur especially in areas immediately under the starting cones or at localized high concentrations of iron where hot, molten NaCl could flow downward and, on solidification, effectively form seals that would hold H₂O. On this basis glass-fiber candles should produce more chlorine than metal-fiber ones, because the relatively low-melting-point glass would be expected to contribute to seal formation.

Concentrations of water as low as 0.1% in the candle mixture may produce significant amounts of chlorine, as shown by sample 6E of Table 6. Localized high concentrations of iron metal, especially from iron powder, would be expected to give intermittent halogen formation on the basis of the "seal mechanism;" however, even large concentrations of water (4E, Table 6) would not necessarily produce proportionally large concentrations of chlorine, since the bulk of the water can evaporate from the reaction zone readily.

Table 6
Effect of Entrapped Water on Available Chlorine

Sample	Remarks	NaClO (wt-%)	NaClO ₂ (wt-%)	Available Chlorine (ppm by volume)
1E	0.1% H ₂ O, open	0	0	0
2E	0.7% H ₂ O, open	0	0	0
3E	1% H ₂ O, open	0	0	0
4E	2% H ₂ O, open	0.0035	0	20
5E	0.1% H ₂ O, 20% Fe, open	0	0.001	5
6E	0.1% H ₂ O, sealed (1)	0	0.009	40
1F	0.3% H ₂ O, open	0.0015	0	80
2F	0.3% H ₂ O, 33% NaCl-open	0.0015	0	80
3F	4% H ₂ O, open	0.011	0.0025	70
4F	2% H ₂ O, sealed (1) ignition on top	0.0015	0.016	80
5F	2% H ₂ O, sealed (1) internal ignition	0	0.036	160
6F	2% H ₂ O, sealed (2) internal ignition	0.022	0.047	325
7F	0.1% H ₂ O, sealed (2) internal ignition	0.0075	0	40
8F	2% H ₂ O, 33% NaCl sealed (1) ignition on top	0.009	0.0025	50

E - 50-gram candle mix--88% NaClO₃, 4% Fe, 4% glass fiber, 4% BaO

F - 44-gram reagent NaClO₃; BaO or BaO₂ not present

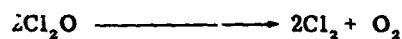
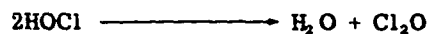
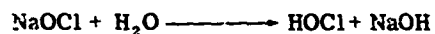
(1) External surface (top) sealed with fused NaClO₃, M.P. 250°C

(2) External surface (top) sealed with fused NaCl, M.P. 800°C

Samples E and F powdered. Combustion 30 seconds, stopped by quenching.

The following reaction mechanisms, based on results of Tables 5, 6, and 7, are postulated:

NaOCl decomposition (11):



NaClO₂ decomposition (12a, 13):

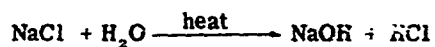


Table 7
Chlorine Evolved During NaClO_3 Decomposition in the
Presence of Trace Amounts of Water*

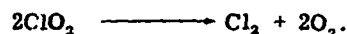
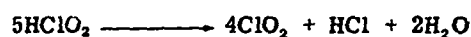
NaClO_3 (grams)	Description†	Detected Cl_2 (ppm)‡
9.5	Pyrex wool plug not used; 50-ml steel crucible	14
0.95	Pyrex wool plug not used; 50-ml steel crucible	11
0.95	Plug not used, crucible very rapidly brought to a yellowish red ($\sim 1100^\circ\text{C}$)	160
2.85	Plug tamped loosely, NaClO_3 in pyrolysis tube	30
0.45	Plug loose sample in pyrolysis tube	17
0.45	Plug firmly tamped in pyrolysis tube	191
1.90	Plug firmly tamped in pyrolysis tube	151
0.45	Plug firmly tamped in pyrolysis tube	405
0.45	Plug firmly tamped in pyrolysis tube	249
0.45 + 0.2 g NaCl	Plug firmly tamped in pyrolysis tube	249
0.45 + 0.2 g Fe powder	Plug firmly tamped in pyrolysis tube	388
0.45 + 0.3 g Fe powder	Plug loose§	18

*The NaClO_3 after 7 days at 115°C retained 0.16% H_2O .

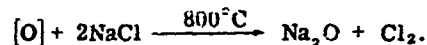
†Pyrex wool and NaClO_3 holder (steel crucibles or combustion tubes) dried at 220°C for 5 hours. Decomposition temperature 800°C - 1000°C .

‡1/a Dräger chlorine detector tubes used. PPM by volume. Cl_2 fraction through Dräger tube 1/10 of total Cl_2 ; results converted to total O_2 flow.

§Pyrex wool plug in all runs, unless stated otherwise, was in contact with the NaClO_3 .



Salt decomposes at high temperature (12b) or in presence of certain catalysts:

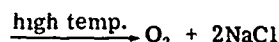
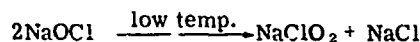


The latter is not believed to occur to any significant extent during combustion unless localized high-temperature spots exist. (This condition may occur in burning Al, Mg, or B candle compositions.) Certain catalysts, such as $\text{K}_2\text{Cr}_2\text{O}_7$, favor Cl_2 production (14).

Trace-metal chlorates or perchlorates, such as $\text{Mg}(\text{ClO}_4)_2$, also could be cited in potential mechanisms for halogen formation, but the concentration of cation impurities found by spectrographic analysis were too low to be significant.

The mechanism of halite formation is postulated as follows:

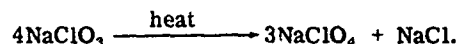




If the NaClO_3 molecule were an oxygen acceptor rather than NaCl , the following reactions would take place:



The mechanism could also be:



The former has experimental verification (10), thus providing an explanation for the presence of NaClO_2 at high temperatures.

CONCLUSIONS

A departure from the present technique for fabrication of chlorate candles has been investigated and found to be at least competitive and not superior. The present method is to add 3% water to the chemical mixture just before pressing at ambient temperature, and then to drive out as much of this water as possible by oven heating. The new method is to heat the mixture by rf induction and then to press in a hot die. The addition of the heating step results in a candle having considerably higher density and, consequently, more oxygen per unit volume. The candle also has greater resistance to cracking caused by handling or thermal stress during combustion. By this method, high-density candles (2.5 g/cc) have been produced which yield the equivalent of 200 cu ft of oxygen (STP) from 490 cu in. of candle volume. In addition, the production of chlorine is reduced significantly.

1. Molded candles are best formed at temperatures from 225° to 245°C at pressures from 10,000 to 5000 psi, respectively.
2. A double-action ram can be used; with a single-action ram, successive pressing increments should be used, as portions of the mix are added, to avoid uneven distribution.
3. A metal fiber, equivalent to grade 00, and length approximately 1 mm, should be used for better agglomeration and combustion.
4. A double-screen procedure should be used for better fiber incorporation. Two screens were used: 6 mesh and 10 mesh. However, two 6-mesh screens can be used.
5. Use of a glass-wool blanket or oven annealing will help to avoid thermal stresses on cooling.
6. If the mixture is heated in batches, avoid long heating periods if possible, in order to reduce recrystallization of the sodium chlorate and fusing of the mixture. A glass cloth laid in the bottom of the pan can be employed for facilitating removal of the semifused candle mixture from the pan.
7. If extrusion processes are employed in candle fabrication, incorporation of 0.5 wt-% potassium perchlorate may be of advantage in prevention of agglomeration of the iron fibers. The potassium perchlorate may also act as a combustion moderator, with a leveling effect on the production of oxygen.

RECOMMENDATIONS

Whether it will be feasible to switch from the presently used candle type to the hot-pressed variety depends on the economics of mass production. The cost per unit volume of oxygen must not be further increased; there is much chafing at the present cost by the submarine operators. It is recommended strongly that contract bids be sought from potential suppliers of hot-pressed candles for production of a pilot quantity of such candles, to be evaluated in the laboratory and on a submarine. A reasonably accurate determination of production costs should be specified in such a contract.

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UNCLASSIFIED Naval Research Laboratory. Report 5732. CHLORATE-CANDLE FABRICATION BY HOT PRESSING, by P.R. Gustafson, S.H. Smith, Jr., and R.R. Miller. 18 pp. and figs., January 23, 1962. An investigation has shown that there are distinct advantages to manufacturing chlorate candles by a hot-pressing technique, omitting water as a binder. Small quantities of water trapped in a chlorate candle can result in the production of significant quantities of chlorine. By the hot-pressing method, high-density candles (2.5 g/cc) have been produced which yield the equivalent of 200 cu ft of oxygen (STP) from 490 cu in. of candle volume. UNCLASSIFIED (over)	1. Oxygen candles - Production - Physical factors I. Gustafson, P.R. II. Smith, S.H. III. Miller, R.R.	UNCLASSIFIED Naval Research Laboratory. Report 5732. CHLORATE-CANDLE FABRICATION BY HOT PRESSING, by P.R. Gustafson, S.H. Smith, Jr., and R.R. Miller. 18 pp. and figs., January 23, 1962. An investigation has shown that there are distinct advantages to manufacturing chlorate candles by a hot-pressing technique, omitting water as a binder. Small quantities of water trapped in a chlorate candle can result in the production of significant quantities of chlorine. By the hot-pressing method, high-density candles (2.5 g/cc) have been produced which yield the equivalent of 200 cu ft of oxygen (STP) from 490 cu in. of candle volume. UNCLASSIFIED (over)	1. Oxygen candles - Production - Physical factors I. Gustafson, P.R. II. Smith, S.H. III. Miller, R.R.
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