Industrial trial to produce a low clinker, low carbon cement


a. Center for the Research and Development of Structures and Materials (CIDem), Central University “Marta Abreu” of Las Villas (UCLV), (Villa Clara, Cuba)
b. Siguaney Cement Factory, Cement Enterprise Group (GECEM) (Siguanea, Sancti Spíritus, Cuba)
c. Technical Center for the Development of Construction Materials, Ministry of Construction, (La Habana, Cuba)
d. École Polytechnique Fédérale de Lausanne (EPFL), (Lausanne, Switzerland)

* f.martirena@enet.cu

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ABSTRACT: A preliminary assessment of conditions for the industrial manufacture of a new cementitious system based on clinker-calcined clay and limestone, developed by the authors, referred as “low carbon cement” is presented. The new cement enables the substitution of more than 50% of the mass of clinker without compromising performance. The paper presents the follow-up of an industrial trial carried out in Cuba to produce 130 tonnes of the new cement at a cement plant. The new material proved to fulfill national standards in applications such as the manufacture of hollow concrete blocks and precast concrete. No major differences either in the rheological or mechanical properties were found when compared with Portland cement. Environmental assessment of the ternary cement was made, which included comparison with other blended cements produced industrially in Cuba. The new cement has proven to contribute to the reduction of above 30% of carbon emissions on cement manufacture.

KEYWORDS: Cement; Calcined clays; Energy; Emissions; Production

RESUMEN: Impacto del uso de un cemento de bajo carbono en la mejora de la sostenibilidad de la producción de cemento. Se presenta la evaluación preliminar de las condiciones de fabricación industrial de un nuevo sistema cementicio a partir del empleo de clíinker; arcillas calcinadas y piedra caliza; desarrollado por los autores; denominado “cemento de bajo carbono”. El nuevo cemento posibilita la reducción de más de un 50% de la masa de clíinker; sin comprometer el comportamiento del material. El presente trabajo presenta el monitoreo de la producción industrial en una planta en Cuba; de 130 t del nuevo cemento. El cemento obtenido cumple con las regulaciones nacionales de calidad y su empleo tiene similar rendimiento que el cemento Portland para la producción de bloques y hormigón de 25 MPa. Se realiza el análisis de impacto ambiental del cemento ternario mediante la comparación con otros cementos producidos industrialmente. El nuevo cemento puede contribuir a la reducción de más del 30% de las emisiones de CO₂ asociadas a la manufactura de cemento.

PALABRAS CLAVE: Cemento; Arcillas calcinadas; Energía; Emissions; Producción

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1. INTRODUCTION

The amount of cement produced in 2011 was 3.6 billion tonnes (1). If average content of cement per m³ of concrete ranges around 250 kg of cement; approximately 14 billion tonnes of cement were made only in this year; this is equivalent to approximately 2 m³ of concrete for every inhabitant of planet Earth. No other material is produced in these massive quantities (2).

Approximately 5–9% of anthropogenic total emissions of CO₂ are related to the concrete production. This is caused by the massive production and use of cement in the construction sector; 85% of total emissions are associated with cement manufacture; thus its contribution is approximately 3–5% of global CO₂ emission (3, 4). Any improvement in cement manufacture may have a great impact on the reduction of CO₂ released to the atmosphere.

Tackling the urgent housing problems in developing countries prompts for the massive availability of construction materials. The demand of concrete emissions are associated with cement manufacture (5). Further; the availability of suitable SCMs is limited and it is closely related to the cement.

The amount of cement produced industrially in Cuba. The two SCMs used in the new system; calcined clay and limestone have a higher availability than other SCMs. Low grade kaolinite clay has proven to be a suitable alternative to the pure kaolinite metakaolin (MK) currently used by the industry (10). Low grade kaolinite clay reserves are huge and are better geographically distributed than pure kaolinite clay deposits. The size of limestone reserves is in the same order of magnitude as current world cement production (7).

This article presents the results of an industrial trial carried out at a cement plant in Cuba for the production of the new cement at industrial scale. Comparisons between industrial and lab results were made. Further; a preliminary environmental assessment of the production of the new cement was done; which included a comparison with cements produced industrially in Cuba.

2. CLINKER SUBSTITUTION RATE AT BLENDED CEMENTS VS. TERNARY BLEND CEMENTS

Pozzolans have been extensively used as SCM to substitute clinker in cement production. They react as described below –for silica rich pozzolanic systems, (11, 13), [1]:

\[
S + 1.5CH + 2.8H \rightarrow C_1S_{1.8}H_{2.8}
\]

silica+calcium hydroxide+water = calcium silicate hydrate \[1\]

The amount of clinker than can be substituted by pozzolans depends on the availability of calcium hydroxide produced during cement hydration. In pure silica pozzolanic systems; for substitution ratios above 15% wt.; calcium hydroxide produced during hydration is not sufficient to react with all the pozzolanic material (see Table 1); and thus; the unreacted material performs like a filler (13). Higher replacement levels can compromise the mechanical properties of the binder as kinetics of the pozzolanic reaction is usually slower than in pure clinker systems. For this reason most of cement producers
limit the clinker replacement up to approximately 25–35% wt.

Metakaolin (MK) ($\text{Al}_2\text{Si}_2\text{O}_7$) is a highly reactive pozzolan produced through the calcination of clays rich in kaolinite mineral. The reactivity of MK depends on several factors; such as temperature; rate of heating and cooling regimen. The optimal temperature of calcination is between 700–800 °C; although the dehydroxylation of the clay is known to begin at 500 °C (14).

The pozzolanic reaction of MK with the calcium hydroxide (CH) in pastes is described as follows (15, 16) [2]:

$$\text{AS}_2 + 5\text{CH} + 3\text{H} \rightarrow \text{C}_4\text{A}_4\text{H}_{13} + 2\text{CSH}_2$$

[2]

Metakaolin is rich in alumina; so its use as SCM increases the volume of new alumina phases in the system. If calcium carbonate is introduced in the system through an external source; for example; limestone; the alumina phases react with it to form the following compounds (8), [3, 4]:

- **Hemicarboaluminate** $3\text{CaO} \cdot \text{Al}_2\text{O}_3 \cdot 0.5\text{Ca}(\text{OH})_2 \cdot 0.5\text{CaCO}_3 \cdot 11.5\text{H}_2\text{O}$ [3]

- **Monocarboaluminate** $3\text{CaO} \cdot \text{Al}_2\text{O}_3 \cdot \text{CaCO}_3 \cdot 11\text{H}_2\text{O}$ [4]

Based on this principle; it is possible to replace a mass of clinker by a similar mass of MK and calcium carbonate mixed in a 2:1 ratio; respectively; to form hydration products; which are able to fill the pore system of the cement matrix and contribute to the strength. Experimental laboratory results (8) show that it is possible to replace up to 45% wt. of clinker without compromising compressive strength of the material. For 60% of replacement; 90% of strength -related to plain cement- can be reached. The alumina phases react faster; so the early strength is not substantially affected (9).

### 3. LOW CARBON CEMENT

The low carbon cement developed is produced by using the ternary systems of clinker-calcined clays and limestone described above. Medium purity kaolinite clay has proven to be a good alternative to MK in this system; thus increasing the availability and reducing production cost of the cement. The limestone introduced to the system is not calcined; thus no extra CO₂ is emitted to the environment (8, 9).

There are already reports of such an approach for ternary Portland limestone blends with alumina rich pozzolans. De Weert et al. (17) and Moesgaard et al. (18) have worked in fly ash-limestone-Portland cement systems; and they have reported an increase of the mechanical properties caused by the synergy between the fly ash and limestone; which favors the formation of carboaluminate phases that provide a beneficial contribution of the performance of the system. Antoni et al. (8) have worked on similar systems; and the synergy established between the Metakaolin and limestone; as well as its contribution to improving the mechanical properties of cementitious systems with high level of clinker substitution has been proven.

Figure 1 a) compares the different alternatives for blended cements and ternary blends at 7 days. The ternary blend cements; with higher clinker substitution rates achieve higher early strength than ordinary blended cements produced with different sources of pozzolan. This trend remains at later ages as presented in Figure 1 b).

### 4. PROOF OF CONCEPT: INDUSTRIAL TRIAL FOR THE MANUFACTURE OF LOW CARBON CEMENT (LCC) IN CUBA

The very promising results for ternary blends were achieved only at lab scale; under strictly controlled conditions. To scale up this process other considerations have to be taken into account. Significant differences between the procedures used for clay calcination and grinding at lab and industrial scale can take place. A full scale industrial trial was carried out for the production of bulky amounts of cement under real conditions. The Cuban cement industry designated the cement factory *Siguaney* for the industrial trial.

The target ternary cement should have clinker content around 50%. The trial included the calcination of 110 tonnes of medium grade kaolinitic clay; mixing
and homogenizing of the calcined material with limestone in a 2:1 ratio; and co-grinding of the synergetic materials with clinker and gypsum.

For the trial; clay from the clay deposit Pontezuela was selected. The material is classified by the authors as a medium grade kaolinite clay; with an average

Figure 1. Compressive strength of blends made with different SCM and different clinker factors; results are presented for (a) 7 days (b) 28 days (8, 19–22).
content of kaolinite of 48.6%; measured by thermogravimetric analysis (TGA). Chemical composition for samples of the clay was assessed aided by X-ray fluorescence (XRF); Table 2 presents the results. X-ray diffraction (XRD) confirmed the presence of companion mineral such as quartz and iron.

Calcination implied the modification of a wet process rotary kiln—regularly used for clinker production—in order to calcine the material on dry conditions (Figure 2). The clay was fed to the kiln and then heated to 750 °C; temperature chosen as optimal for calcination of the material (16, 23, 24).

After the calcination 90 tonnes of calcined material were obtained; the loss of material was associated with moisture content of the original clay; as well as the chemically bound water on the structure of the clay. The calcined material was stored in five heaps.

The quality of calcination was assessed through the dehydroxylation of the calcined clay. The complete dehydroxylation is achieved when all OH groups are released during calcination; and the peak associated with this in TGA disappears. Figure 3 presents the individual results for each of the five heaps of calcined material evaluated. All heaps have been completely dehydroxylated; thus indicating that the material has been fully activated.

The pozzolanic reactivity of the calcined material was assessed through the compressive strength of standardized mortars; in which 30% wt. of cement is replaced by the pozzolanic material; following the protocol of Fernández and Antoni (8, 14). Two reference series were prepared with 100% of ordinary Portland cement (OPC) and cement with 30% wt. pozzolanic material calcined under optimal conditions at the laboratory. Figure 4 presents the results of compressive strength of standardized mortars at 3, 7 and 28 days. The reactivity of the material calcined in the rotary kiln (average of batches) proved to be similar to that of the material calcined at the lab; thus indicating that the industrial calcination was successful.

Grinding was made under industrial conditions; by using a ball mill with a double chamber grinding system (see Figure 5). In order to avoid high specific surface; it was decided to grind the material rather on the coarse side. Gypsum was adjusted to optimize the reaction of the alumina phase (8). The original gypsum used had a low purity; thus the relatively large proportion in the cement composition; but the total SO3 content was adjusted to fulfill cement standards. Finally; grinding parameters were set as: 10–12% retained in the 90 µm sieve; specific surface between 4000–5000 cm²/g and SO3 up to 3.0%.

Grinding was completed in 8 hours. Samples of the material were taken every approximately 30 minutes.

### Table 2. Chemical composition of representative samples of Pontezuela clay

<table>
<thead>
<tr>
<th>Oxides</th>
<th>SAMPLE 1</th>
<th>SAMPLE 2</th>
<th>SAMPLE 3</th>
</tr>
</thead>
<tbody>
<tr>
<td>SiO2</td>
<td>54.7</td>
<td>54.2</td>
<td>55.0</td>
</tr>
<tr>
<td>Al2O3</td>
<td>27.8</td>
<td>28.2</td>
<td>26.0</td>
</tr>
<tr>
<td>Fe2O3</td>
<td>12.1</td>
<td>12.3</td>
<td>13.4</td>
</tr>
<tr>
<td>CaO</td>
<td>1.7</td>
<td>1.7</td>
<td>1.8</td>
</tr>
<tr>
<td>MgO</td>
<td>0.9</td>
<td>0.9</td>
<td>1.0</td>
</tr>
<tr>
<td>SO3</td>
<td>0.0</td>
<td>1.4</td>
<td>0.7</td>
</tr>
<tr>
<td>Na2O</td>
<td>0.3</td>
<td>0.3</td>
<td>0.3</td>
</tr>
<tr>
<td>K2O</td>
<td>1.5</td>
<td>1.6</td>
<td>1.6</td>
</tr>
<tr>
<td>TiO2</td>
<td>0.8</td>
<td>0.8</td>
<td>0.8</td>
</tr>
<tr>
<td>P2O5</td>
<td>0.2</td>
<td>0.1</td>
<td>0.2</td>
</tr>
<tr>
<td>MnO2</td>
<td>0.0</td>
<td>0.0</td>
<td>0.0</td>
</tr>
<tr>
<td>Cr2O3</td>
<td>1.6</td>
<td>–</td>
<td>–</td>
</tr>
<tr>
<td>LOI</td>
<td>10.4</td>
<td>10.3</td>
<td>9.8</td>
</tr>
<tr>
<td>Humidity</td>
<td>3.5</td>
<td>6.1</td>
<td>2.9</td>
</tr>
</tbody>
</table>

Figure 2. Direct feeding of the clay to the rotatory kiln.

Figure 3. Thermo gravimetric analysis of the clay calcined in the rotatory kiln.
Table 3 presents the chemical composition of the material obtained and the average proportion of the components.

The final cement was characterized following the protocol established for blended cements in Cuban standards (25–30). Excess grinding of some softer materials through the interaction with other harder ingredients can be produced during the co-grinding process. This can have an influence on the grain size distribution of the cement; and it could eventually increase water demand of the mix (31). Results of the physical and mechanical tests are presented in Table 4.

5. INDUSTRIAL USE OF THE EXPERIMENTAL CEMENT PRODUCED IN THE INDUSTRIAL TRIAL

The industrial trial enabled the technical team to sound out the reaction of the construction and manufacturing industry to this new binder. Batches of the cement produced were distributed among builders and building material manufacturers; and their use was strictly supervised by the technical team. Potential customers were asked to use the new experimental cement in the same proportions as they usually use the Portland cement.

The trial focused on two main cement applications: (1) manufacture of hollow concrete blocks having size 500×200×150 mm; produced on a semi-automated vibro-compacting machine; and (2) manufacture of 25 MPa precast concrete elements at a prefabrication plant in Cuba. Table 5 presents mix proportions used for both applications. Mix design was accredited by the laboratory of the National Enterprise for Applied Research; in Cuba; following the regulations of Cuban standards (32–36).

Hollow concrete blocks were produced under standard manufacturing conditions; with a 1:1 cement substitution by the new cement. The quality of the blocks was assessed through the Cuban standard NC 247:2010 (37). This standard requires fulfillment of compressive strength and water absorption. Table 6 presents the results of the evaluation of concrete blocks made with the low carbon cement; as well as the reference blocks made with ordinary Portland cement. The experimental blocks met the standard's requirements for compressive strength and water absorption; thus indicating that the new cement can replace Portland cement in this kind of industrial application.

Several cubic meters of 25 MPa concrete were cast under standard manufacturing conditions; with a 1:1 cement substitution by the new cement. Quality of the precast elements produced was assessed aided by the Cuban standards (32–36). Table 7 presents the results of compressive strength of both the experimental and normal concretes cast.
for the trial. Both mixes exceed the 28 day strength prescribed; and again; no major differences in rheology were observed; thus indicating that the new cement can replace Portland cement in this kind of industrial application.

6. ASSESSMENT OF THE ENVIRONMENTAL IMPACT OF THE EXPERIMENTAL CEMENT PRODUCED AT THE INDUSTRIAL TRIAL

Blended cements with clinker substitution up to 30% enable reduction of approximately 15–20% of the CO₂ emissions. The new cement formulation presented in this paper enables to increase clinker substitution to 50% without compromising performance; this represents a reduction of around 30% of the CO₂ emissions associated to the cement manufacture; as Table 8 presents. A preliminary assessment of the environmental impact was made; coupled to the industrial trial at cement factory Siguaney; with the aim of shedding light on the viability of such production from the environmental viewpoint.

Siguaney’s clinker manufacturing facilities consist only of wet-process rotary kilns. Because of its low efficiency; the factory is no longer using its clinker; and the clinker used is transported through railways from the cement plant Cienfuegos; located 100 km and equipped with a dry process rotary kiln of high efficiency. The production strategy also foresees clay calcination at Siguaney in a retrofitted wet process cement kiln. This could enable

<table>
<thead>
<tr>
<th>Material</th>
<th>Retained 4900 Sieve (%)</th>
<th>Consistency (%)</th>
<th>Setting time</th>
<th>Compressive strength (MPa)</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td></td>
<td></td>
<td>Initial (min)</td>
<td>Final (hr.)</td>
</tr>
<tr>
<td>LCC</td>
<td>12.0</td>
<td>25.0</td>
<td>135</td>
<td>2.9</td>
</tr>
</tbody>
</table>

| Table 4. Results of physical and mechanical test of the industrial low carbon cement |

| Table 5. Mix proportions used in concrete manufacture |

<table>
<thead>
<tr>
<th>For 1 m³</th>
<th>Gravimetric mix proportion (kg)</th>
<th>Mix proportion (m³)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Materials</td>
<td>LCC</td>
<td>Sand “El Purio” quarry</td>
</tr>
<tr>
<td>Hollow block 150 mm</td>
<td>Concrete 25 MPa</td>
<td>Hollow block 150 mm</td>
</tr>
<tr>
<td>LCC</td>
<td>300</td>
<td>654</td>
</tr>
<tr>
<td>Sand “El Purio” quarry</td>
<td>360</td>
<td>–</td>
</tr>
<tr>
<td>Powder “Palenque” quarry</td>
<td>–</td>
<td>–</td>
</tr>
<tr>
<td>Aggregates 5–13 mm “El Purio” quarry</td>
<td>–</td>
<td>–</td>
</tr>
<tr>
<td>Aggregates 19–10 mm “Palenque” quarry</td>
<td>–</td>
<td>–</td>
</tr>
<tr>
<td>Water (L)</td>
<td>–</td>
<td>–</td>
</tr>
<tr>
<td>Superplasticizer Dynamon SX-32 (L)</td>
<td>–</td>
<td>–</td>
</tr>
<tr>
<td>w/c batch</td>
<td>–</td>
<td>–</td>
</tr>
<tr>
<td>w/c effective</td>
<td>–</td>
<td>–</td>
</tr>
<tr>
<td>Designed slump</td>
<td>–</td>
<td>–</td>
</tr>
</tbody>
</table>

<table>
<thead>
<tr>
<th>Dimensions hollow blocks (mm)</th>
<th>Average compressive strength at 7 d (MPa)</th>
<th>Average compressive strength at 28 d (MPa)</th>
<th>Performance</th>
<th>% absorption</th>
</tr>
</thead>
<tbody>
<tr>
<td>500×200×150</td>
<td>3.3</td>
<td>5.9</td>
<td>2.0</td>
<td>5.6</td>
</tr>
<tr>
<td>Specification</td>
<td>4.0</td>
<td>5.0</td>
<td>–</td>
<td>–10</td>
</tr>
</tbody>
</table>

*Relation between the compressive strength obtained at 28 days in kg/cm² and the cement consumption.

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using the existing infrastructure for production in Siguane–calcining clay; grinding cement; storing; etc. - and yet achieve some degree of efficiency in the process.

Clay calcination is foreseen in two alternative scenarios:
1. Short term: Production of calcined clays in using an existent wet process rotatory kiln in Siguane factory.
2. Long term: Production of calcined clays in an industrial retrofitted calciner to be set in Siguane factory; operating with dry process.

CO₂ emissions during cement manufacture were calculated by dividing the productive process in two phases: (i) clinker and clay calcination; and (ii) mixing and grinding of the clinker and additions. During the calcination process; three main emission sources can be defined: a) Fossil fuels combustion; b) chemical decomposition of calcium carbonate (CaCO₃) and magnesium (MgCO₃) and c) electricity consumption. The methodology to estimate the CO₂ emissions followed the recommendations of the Intergovernmental Panel for Climate Change; 2006 (IPCC) (38); whereas clinker (CK) production is multiplied by an emission factor. So; the CO₂ emissions caused by the energy consumption (a and c); are calculated using the following formula [5]:

\[
\text{CO}_2 \text{ emissions} = \text{Emission factor} \times \text{Consumption index} \ [5]
\]

Where:
- CO₂ emissions: kg CO₂/tonne CK-Calcined clay
- Emission factor: kg CO₂/unit of energy used
- Consumption index: unit of energy used/tonnes of CK-Calcined clay.

The dry process clinker production operates with pet-coke and the electricity is supplied by the National Energy System (NES) as energy sources. Table 9 presents the emissions associated to the energy consumption on the first stage of cement manufacture; mainly clinker production.

For clay calcination at cement plant Siguane; crude oil was used and electricity was supplied by the National Energy System. Table 10 displays the emissions associated to energy consumption during clay calcination in the short and long term scenarios.

The emissions caused by the chemical decomposition of CaCO₃ y MgCO₃ contained in raw materials (grey limestone and clays); may be deduced through stoichiometry calculations with two different approaches:

a) By calculating the differences in the CaO and MgO content of the raw materials before been fed to the kiln and at the exit (CK or calcined clay) (39).

b) By assessing the contribution of each component of the paste to the total CO₂ emissions; through the knowledge of the chemical composition of the minerals used in the materials at the entrance of the kiln and its CaCO₃ and MgCO₃ content.

The use of one or other approach is determined by the data availability; which helps to describe the chemical composition of the material; before and after processing it.

The authors chose to measure the differences between the CaO and MgO content of the raw materials while entering or exiting the kiln. The differences in the oxides content are converted by stoichiometry on the CO₂ released.

The CO₂ emissions due to the decarbonation of limestone were calculated for the conditions of Cienfuegos factory; which is the main clinker supplier for cement production in Siguane. Table 11 presents the calculation of the emissions at the decarbonation of calcium and magnesium carbonate during clinker production at cement plant Cienfuegos.

The CO₂ emissions in the phase of cement grinding; are associated with the electricity consumption in the ball mill; see Table 12. Other sources of emissions are considered negligible and thus discarded.

The final calculation of CO₂ emissions is presented in Figure 6. The low carbon cement produced in non-optimized conditions during industrial trial;
reduces approximately 270 kg CO₂/tonnes in relation to OPC (P-35); this is approximately 31%. Reduction in reference to traditional Cuban blended cement (PP-25) is in the range of 125 kg CO₂/tonne. P-35 and PP-25 are both regularly produced at cement plant Siguaney.

In the projected long term scenario; the total estimated emissions for low carbon cement achieve reductions higher than 300 kg CO₂/tonne in relation to P-35 and 200 kg CO₂/tonne in relation to PP-25. This represents more than 35% of reduction compared to business as usual practice.

Figure 7 presents emissions and compressive strength at 28 days of LCC produced at the industrial trial; compared with reference values of cements P-35 and PP-25. This illustrates the idea that LCC, despite its very low clinker content which leads to low carbon emissions; does not compromise performance of cement; even compared with other cements produced industrially at the cement plant.

7. CONCLUSIONS

- There are alternatives available to move the current boundaries of clinker substitution for the production of blended cements through the use of ternary systems based on clinker, calcined clays and limestone. The principle behind this
The proposal is the synergy between calcined clay and limestone; which allows increasing the reactivity of the SCMs and reduces clinker factor. This system is based on the use of medium grade kaolinitic clay; and only small changes should be made to the production process. Reserves of medium grade clay and limestone are much higher than any other SCM.

The industrial trial to produce cement with a clinker factor of 50% at the industrial scale has proven that the new system is very robust; while even in non-optimized conditions acceptable results have been achieved in terms of performance of the resulting material as cement; as well as in its applications in concrete.

The new cementitious system could enable reduction on the emissions associated to the cement manufacture in the range of 25–35% related to business as usual practice. This reduction is based on replacing clinker; which is the main CO₂ releaser; by a combination of materials whose emissions are negligible compared to clinker.

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Figure 6. CO₂ emissions associated to LCC production with 45% of SCM and compared to three reference cements P-35 and PP-25.

Figure 7. Relation between emissions of CO₂/ton cement and associated compressive strength of P-35; PP-25 and LCC produced in industrial trial.


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