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TRANSIENT BOILING OF LIQUEFIED CRYOGENS ON A WATER SURFACE

II. LIGHT HYDROCARBON MIXTURES

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Abstract—Mixtures of light hydrocarbons characteristic of liquefied natural gas were boiled on a water surface and the rate of vaporization measured. Heat fluxes were significantly higher than measured for pure liquid methane even when mole fractions ethane, propane, or *n*-butane were under 0·01. As with pure methane, the rate of vaporization increased during the course of an experiment unless a continuous, thick ice layer formed. Initial liquid water temperatures ranged from 6 to 60°C and spills of LNG from 0·21 to 0·83 g/cm^2 were made. For mixtures of methane and heavier hydrocarbons, where the mole percent of the latter components was greater than 2 per cent, the water surface temperature rapidly dropped to the cryogen temperature, but little change in temperature was noted a few mm below the surface. No significant vapor superheat was noted. Ice formed readily at the interface and the increase in heat flux was explained by postulating a shift from film to nucleate boiling. The boiling mixtures often foamed and photographs taken at the interface revealed that very small vapor bubbles formed early in a run.

NOMENCLATURE

A,	slope of lines in Figs. 2–7;
B,	constant in equation (1);
M',	mass of liquid cryogen spilled;
t,	time.

INTRODUCTION

IN THE previous paper [1] experimental rate data were presented for pure liquefied nitrogen, methane, and ethane boiling on a water surface. In the present paper, we extend the study to mixtures of light hydrocarbons characteristic of liquefied natural gas (LNG).

The boiling fluxes reported here should be of value to those charged with preparing hazard analyses or environmental impact statements wherein hypothetical accidents are assumed for LNG tankers or barges in harbors or inland waterways. The rate of vapor generation must be known before one can assess downwind vapor concentrations and thereby delineate the extent of the flammable vapor cloud.

The study is also of interest in a more general sense as few experimental investigations have been made wherein a volatile liquid mixture boils while in contact with a hotter, less volatile liquid. For the specific case of light hydrocarbon mixtures in contact with water, only three previous reports are available [2–4] and, even here, the results are largely qualitative in nature.

Boiling heat fluxes for mixtures are often quite different from those of the pure components comprising the mixture. As an example, Vos and van Strahlen [5] and van Wijk *et al.* [6] found that the peak nucleate flux for methyl ethyl ketone–water mixtures was about 120 per cent larger than for pure water. At the composition for this maximum flux (4·2 wt% ketone), the bubble sizes were also found to minimize [6, 7]. These interesting results were explained by a model [8] that allowed for the decrease in concentration of the more volatile component in the liquid film around each bubble. The temperature driving force for vaporization in the superheated liquid was, therefore, decreased and smaller bubbles resulted. Scriven [9] has also treated the problem of bubble growth in superheated liquid mixtures.

Hovestreijdt [10] has also considered this maximum heat flux–minimum bubble size-phenomenon and has advanced an alternate theory. He argued that for *positive* systems, i.e. those wherein the more volatile component has the lower surface tension, there is a stabilization of smaller bubbles by a Marangoni effect. LNG is a good example of such a system and, as will be shown later, small additions of heavier hydrocarbons to methane significantly increase the heat flux, though the boiling regime is not clearly nucleate. Other studies dealing with nucleate boiling of mixtures include [11–13].

Sliepcevich *et al.* [14] reviewed the available heat-transfer data for mixtures of light hydrocarbons and state that the boiling of LNG on solid surfaces can be unexpectedly different from the pure components or binaries. Based on only scanty data [15], they noted that the nucleate boiling range was greatly expanded compared to the pure light hydrocarbons, the burnout fluxes much higher, and the lowest stable film boiling fluxes at one atmosphere were quite large (~130–150 kW/m^2). At the Leidenfrost point, the temperature difference was over 350°C.

Studies involving binaries of ethane, ethylene, propane, *n*-butane, and *n*-pentane [16, 17] have also shown that the boiling rates of hydrocarbon mixtures can be significantly different from the pure components.

For film boiling of mixtures, few data exist to delineate the effect of composition on heat flux.

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Kautzky and Westwater [18] did find that small additions of Freon 113 to carbon tetrachloride increased the boiling rate some 20 per cent higher than pure CCl_4. Yue and Weber [19] analyzed film boiling of binary mixtures by a two-phase boundary-layer theory and predicted that the boiling flux would increase for mixtures with a higher relative volatility. Experimental data were reported for several organic mixtures and, although there was good agreement between theory and experiment, in no case was there found a disproportionate effect of composition on the heat flux.

Van Strahlen *et al.* [8] carefully studied film boiling of a methyl ethyl ketone mixture (4·2% ketone by weight) with water and reported that, relative to pure water, the heat flux increased by a factor of 1·2–1·8 depending on the heating wire temperature. By the use of high-speed motion pictures of the vapor production rates, they concluded that, for the mixture, only about 50 per cent of the total heat flux resulted in vapor production at the film. For pure liquids, this same value exceeded 95 per cent. It was postulated that there was local exhaustion of the more volatile component at the film interface with a concomitant increase in film temperature. Thus, appreciable conduction heat transfer resulted from the formation of this temperature gradient in the adjacent liquid layer.

EXPERIMENTAL

The apparatus and experimental procedures are described in the previous paper [1]. Hydrocarbon mixture compositions were measured by gas chromatography and the details are available elsewhere [20]. The difficult task of obtaining representative samples from liquid hydrocarbon mixtures was solved by employing a special sampling technique using the bulb shown on Fig. 1. To withdraw a sample, the cannula was first dipped into the liquid and allowed to cool while the bulb was evacuated. Several liquid slugs were allowed to flash vaporize within the bulb, with evacuation between sampling, before gas was removed in the syringe for analysis. Samples were taken from the spill-Dewar flask just prior to the pour on water.

EXPERIMENTAL RESULTS

Twenty spill and vaporization experiments were made with synthetic binary mixtures containing methane and ethane, propane, or *n*-butane. In these tests, the initial water temperature was varied from about 10 to 20°C and the mass spilled from 20 to 30 g. Since the spill area was 77·3 cm^2, this range corresponded to spills of 0·26 to 0·39 g/cm^2. Thirty-one experiments were made with mixtures containing methane, ethane, propane, and *n*- and *i*-butane. The initial water temperature and quantities spilled in these tests ranged from 6 to 60°C and from 16 to 64 g (0·21–0·83 g/cm^2).

In all experiments, temperatures were measured in the water at three depths (which varied from run to run) and in the vapor at two heights. The system mass and each temperature was sampled every second. Complete data for all runs are available elsewhere [20].

LIGHT LNG

Two compositions of *light* LNG were studied. In one, the composition was 98 per cent methane and 2 per cent ethane. In the other, the mixture consisted of 98·2 per cent methane, 1·6 per cent ethane and the remaining 0·2 per cent was made up of propane and trace butanes. All percentages are on a molal basis. The results from experiments with both compositions are discussed together as there was no discernible difference found for the two mixtures. In all cases, the light LNG boiled at a more rapid rate on water than did pure methane. On Fig. 2, we show some data for spills of about 63–64 g (~ 1·9 cm) on water at different initial temperatures. The mass boiled as a function of time was found to plot as a straight line on log–log paper. The runs made at both high initial water temperatures (> 50°C) and at low initial water temperatures (< 10°C)

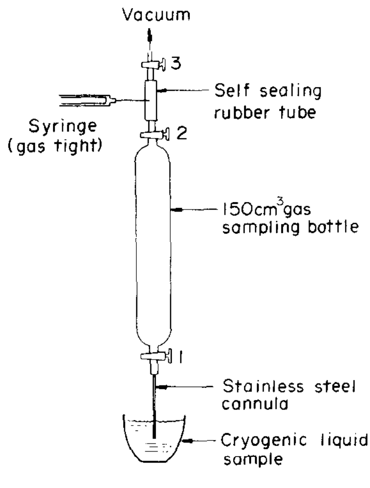


FIG. 1. Apparatus for sampling cryogenic liquids.

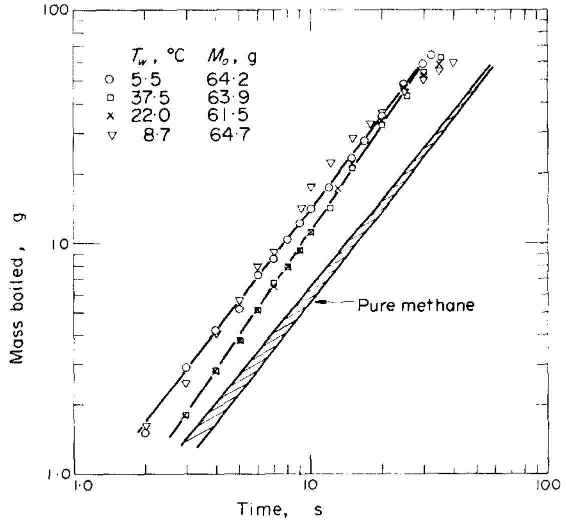


FIG. 2. Boiling of light LNG on water—large spills.

consistently led to more rapid boiling. For comparison, the data for pure methane are shown as a band; this band is the same as that shown in Fig. 3 of the preceding paper [1].

For spills between 20 and 40 g (0·6–1·2 cm), at all water temperatures greater than 10°C, the mass vaporized as a function of time is shown as a narrow band on Fig. 3. Again, the data for pure methane are shown as a reference.

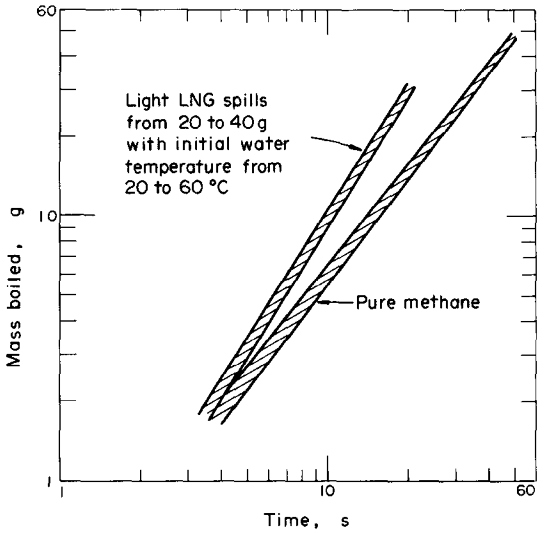
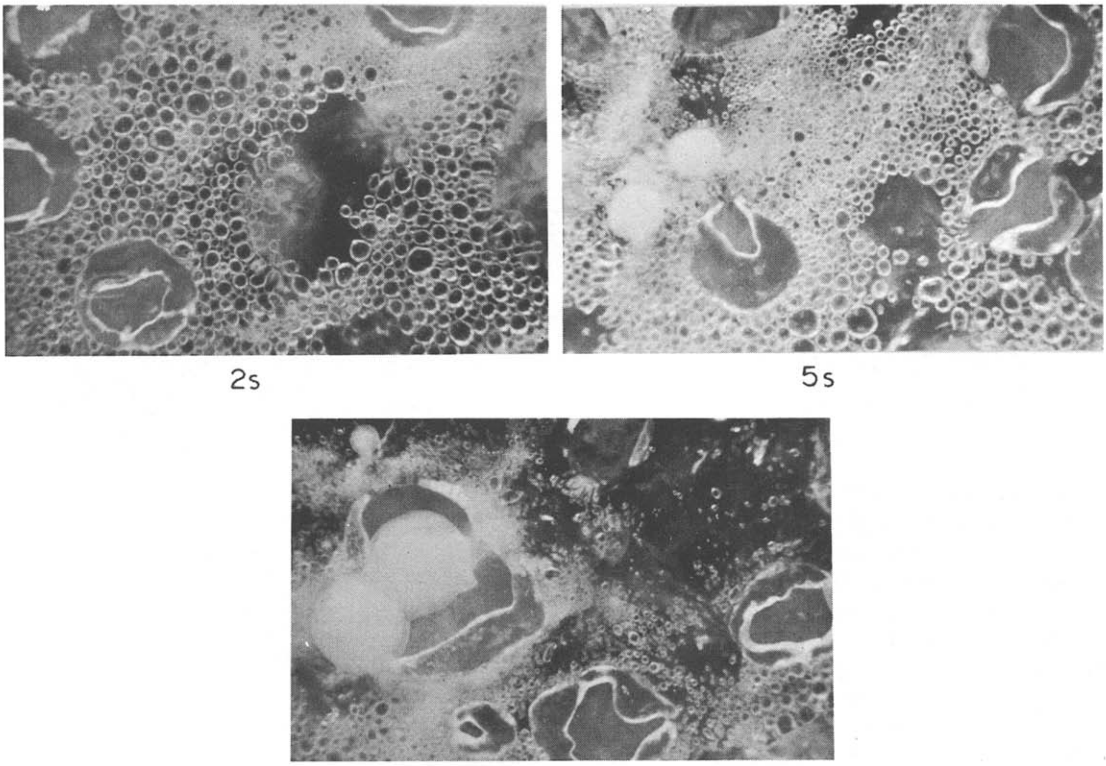


FIG. 3. Boiling of light LNG on water—intermediate spills.

The band for the LNG tests comprises some fourteen different experiments. Though the initial water temperature ranged from 60 to about 10°C, no trends could be found indicating that this variable was important for intermediate size spills. In most tests, some 25–30 g of LNG were spilled on the water; however, there were three tests with greater than 30 g and four with less than 25 g. No tests are shown on Fig. 3 in which less than 20 g were spilled. For the few experiments carried out with smaller spill quantities, the boiling rates were always significantly lower. A similar result was noted in pure component tests [1] and was explained by the supposition that discrete islands of LNG were formed rather than the continuous film assumed in computing experimental heat fluxes.

Temperatures measured a few mm below the water surface showed little change during a run, dropping a few degrees Centigrade by the end of the test. In contrast, temperatures at the water surface quickly dropped to LNG temperatures and remained at this level. For the first few seconds after a spill had been initiated, very small (but numerous) gas bubbles formed at the interface and the LNG foamed. At later times, the number of the smaller bubbles decreased but the average bubble size was always less than those observed with pure components. Three photographs showing the LNG–water interface are presented in Fig. 4. A bimodal size distribution is obvious and the larger bubble size is very similar to that for pure methane (~ 13 mm dia). Note at 5 s, some ice patches are already formed.


2s

5s

9s

FIG. 4. Photographs of light LNG–water interface at various times. Initial water temperature = 18°C. LNG contained 98·2 per cent methane, 1·62 per cent ethane, 0·11 per cent propane and trace higher hydrocarbons.

In summary, with light LNG containing about 98 per cent methane, boiling occurred more rapidly than with pure liquid methane when spills were made on a bounded free water surface. The rate of boiling *increased* with time and foaming occurred, particularly at the start of the test. Surface water temperatures were measured and found to be between − 145 and − 155°C for all tests; in contrast, the temperatures a few mm below the surface showed only a very small, but continuous decrease. Rates were, in general, increased when the initial water temperature was less than 10°C or when large spills were made. Good reproducibility was attained when similar runs were carried out.

HEAVY LNG

Nine spill tests were made with LNG mixtures containing appreciable quantities of ethane and propane. The mole fraction methane ranged from 82 to 89 per cent and the ethane/propane ratios were held to between 4 and 5. Trace quantities of *n*- and iso-butane were present (about 0·2 per cent of each). For each run, a very linear line resulted when the mass lost in boiling was plotted as a function of time on log–log paper. All such lines were parallel and were within the band shown on Fig. 5. Since the mass spilled and initial water temperature also varied from run-to-run, it was not possible to discern any clear trends to separate the effects of these variables. As done in earlier plots, the data for pure methane are also shown.

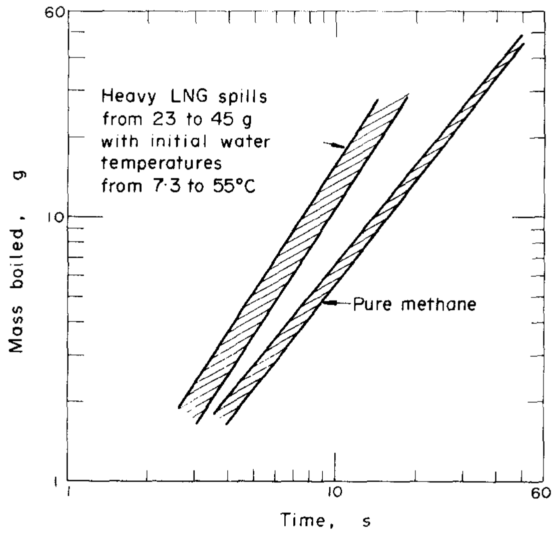


FIG. 5. Boiling of heavy LNG on water.

The heavy LNG band is parallel to, but lies above the light LNG band (Figs. 2 and 3) indicating that increasing the fraction of heavy components leads to more rapid vaporization. As with the light LNG, foaming was noted with the evolution of many very small bubbles. Also, the *surface* liquid water temperature dropped quickly to a value close to LNG temperature, but little change in water temperature could be measured a few mm below the surface.

Not shown in Fig. 5 was the pronounced "tailing" of the mass–time curve in some runs. For all tests carried out with low initial water temperatures, after 20–25 g of LNG had boiled-off, the slope of the log (mass boiled)–log time curve began to decrease and approached a value of about 0·5. One interpretation of this result is that for low initial water temperatures, a continuous solid ice crust formed and the thermal resistance of this layer became important in the later stages of a test. For high initial water temperatures, though ice most certainly formed, the thickness never became large enough to act as a significant resistance.

The exact reason for the *almost* random scatter of the lines for each run within the band shown on Fig. 5 is not known. It is almost as if the subsequent behavior of the boiling liquid were decided by the results after 2 or 3 s. If the very early boiling rate were high, then the run would progress with proportionately higher and higher rates. If, on the other hand, a lower initial rate were measured, then, though the boiling rate would still increase during a run, never would it *catch-up* with the test results for the run that started at a higher level.

METHANE WITH TRACE QUANTITIES OF HEAVIER HYDROCARBONS

Butane

Data taken for a run with 0·16 per cent *n*-butane in methane are shown in Fig. 6. The slope is parallel to that found for pure methane but the displacement is appreciable. At 20 s, for example, about 15 g of pure methane have boiled, but with only the addition of 0·16 per cent *n*-butane, at the same time, about 25 g have vaporized. Temperatures measured near the interface showed an erratic behavior similar to the pure methane spills and temperatures fluctuated around 0°C but never did they drop far below the ice point.

Propane

Experiments were made with 0·15–0·6 per cent propane in methane and the results shown on Fig. 7.

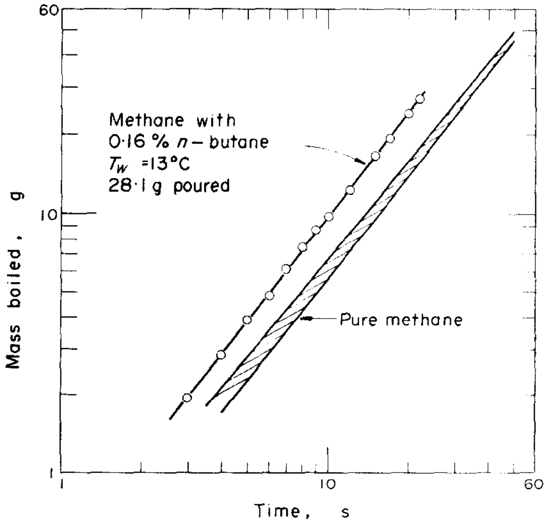


FIG. 6. Boiling of methane with trace *n*-butane on water.

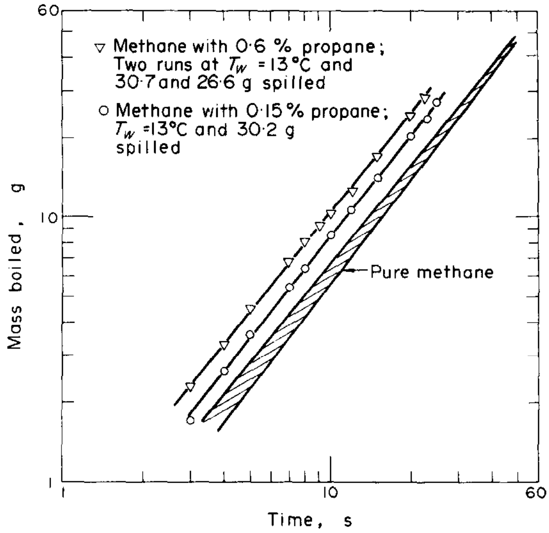


FIG. 7. Boiling of methane with trace propane on water.

The results are similar to those found with butane addition including the unusual behavior of the surface temperatures which fluctuated above and below the ice point.

Other experiments were made with ethane addition but the results were similar to those noted earlier for light LNG at comparable compositions.

DISCUSSION OF LNG RUNS

The phenomenon of boiling LNG upon a water (and ice) surface is obviously very complex. It is difficult to present many unqualified generalizations or conclusions from the data obtained and extensive further studies will be necessary to formulate and verify a model that describes the actual events and would lead to predictive equations.

One of the unexpected results was the high degree of linearity of the data when plotted as in Figs. 2, 3 and 5–7. With M'_0 as the original mass of liquid spilled and M' the mass remaining at any time t, the correlation indicates that

$$(M'_0 - M') = Bt^A. \tag{1}$$

For both light and heavy LNG experiments, A was found to be close to 1·6 whereas in the pure methane and methane with trace heavier hydrocarbons, A was about 1·3. Rearrangement of equation (1) after differentiation yields

$$-\mathrm{d}M'/\mathrm{d}t = A(M'_0 - M')/t. \tag{2}$$

The *rate* of boiling is, of course, proportional to $\mathrm{d}M'/\mathrm{d}t$. $(M'_0 - M')/t$ is the *average* rate of boiling from $t = 0$ to $t = t$. Since A is greater than unity, equation (2) indicates that the boiling rate accelerates. Why this should occur is not at all clear, especially when only two values of A (1·6 or 1·3) were found in this study. In examining the experimental data, the only significant difference noted between runs with different values of A was that, for A to be 1·6, interfacial water temperatures were always close to the LNG temperature whereas, for A = 1·3, the surface temperature remained near 0°C with excursions above and below this value. Except for the case of pure methane, discussed earlier, essentially no vapor superheat was noted.

There are few data with which to compare the results reported here. The Tokyo Gas Co. [2] reported in 1971 that the rate of boiling of LNG on a water surface increased with time and higher rates resulted when the initial water temperature was low. Boyle and Kneebone [4] in 1973 described a few tests wherein LNG ($\sim 95\%\,CH_4$) was spilled on sea water contained in basins 4 and 9 ft^2 in area. Most results were described in a qualitative manner but they also noted that the rate of boiling increased with time and with the amount spilled. Temperatures measured 5 mm below the water surface varied little during most tests. When pools of LNG were less than about 2 mm thick, there was observed a rapid drop in the rate based on the superficial area and this suggested that discrete islands of LNG were forming. Their data also plot as straight lines when shown as in Figs. 2, 3 and 5–7, but the overall rate is slightly less than found here though the slope (A) is higher (1·8–1·9). Their explanation regarding the increase in boiling rate with time focused upon the change from film to nucleate boiling as the surface became progressively covered with ice and the fraction of heavy hydrocarbons increased.

One of the more interesting findings of Boyle and Kneebone was that there was a weight *gain* in the system if the measurements were made *before* the solid surface crust of ice melted. After melting, a net loss of weight was found. The loss could be explained by water loss. Copious white clouds are always observed when cryogens boil on a liquid water surface and this is believed due to small ice fragments entrained in the evolved vapor. The weight gain observation before melting was *not* duplicated in runs with pure liquid methane or nitrogen, and it was suggested that stable heavy hydrocarbon hydrates were formed in the ice crust when LNG was boiled. During melting, these hydrates would decompose as they are unstable or, at least, less stable at ambient temperatures.

If the hypothesis of hydrate formation were true, this could well explain the significant enhancement in boiling rates for LNG compared to pure methane or nitrogen. Further studies are obviously required to verify this concept.

To complete the discussion of the Boyle and Kneebone experiments, it is well to note that they also carried out some unrestricted spill tests in an open pond. Little ice was formed and low boiling rates were reported. This further strengthens the hypothesis that surface ice is necessary to promote nucleate boiling. Thin ice (or hydrates) on a surface of an open pond with waves and wind would readily melt and leave an ice-free interface. (A few restricted area tests with agitation of the water also led to little ice formation and low boiling rates.)

The only other reference to quantitative boiling studies of LNG on water was found in a 1972 Bureau of Mines report [3]. Only average boiling rates were

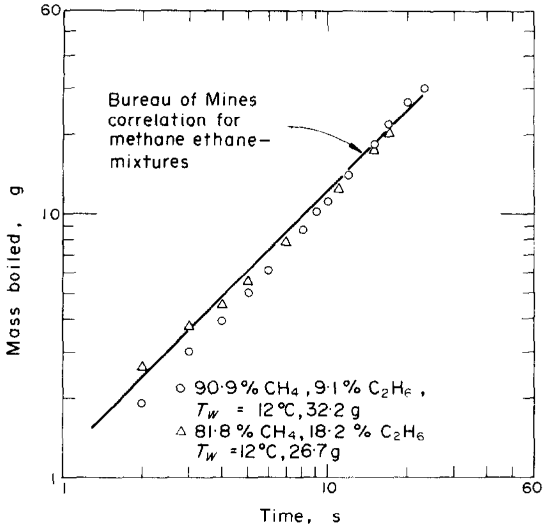


FIG. 8. Boiling of methane–ethane mixtures on water.

reported for the first 20 s following a spill and a value of 0·032 lb/ft^2-s was shown for 2–3 l spills on a 742 cm^2 water surface at 21°C. When converted to the spill area used in the present study (77·3 cm^2), some 24 g of LNG would have been predicted to boil away in the first 20 s. The LNG used by the Bureau of Mines contained from 84 to 88 per cent methane with the remainder ethane and some O_2 and N_2. Almost no propane or higher hydrocarbons were present. The Bureau of Mines' constant rate equation is shown on Fig. 8 and on the same plot, data are shown for two methane–ethane tests made in the current study. Agreement is good though our data clearly indicated an increase in boiling rate with time in contradistinction to the linear (slope = 1) correlation of the Bureau of Mines.

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EBULLITION TRANSITOIRE DES LIQUIDES CRYOGENIQUES A LA SURFACE DE L'EAU. II—MELANGES D'HYDROCARBURES LEGERS

Résumé—Des mélanges d'hydrocarbures légers spécifiques du gaz naturel liquéfié ont été mis en ébullition à la surface de l'eau et la vitesse de vaporisation a été mesurée. Les flux de chaleur étaient nettement plus élevés que ceux mesurés dans le méthane pur liquide même lorsque les fractions molaires d'éthane, propane ou *n*-butane étaient inférieures à 0,01. Comme dans le cas du méthane pur, la vitesse de vaporisation augmentait pendant la durée de l'expérience à moins que ne se forme une couche de glace épaisse et continue. La température initiale de l'eau liquide était comprise entre 6 et 60°C et la nappe de gaz naturel liquifié à été répandue en couches allant de 0,21 à 0,83 g/cm^2. Dans le cas de mélanges de méthane et d'hydrocarbures plus lourds pour lesquels le pourcentage molaire de ce dernier constituant était supérieur à 2 pour cent, la température de surface de l'eau tombait rapidement à la valeur de la température du cryogène sans qu'aucun changement appréciable de température ne soit observé quelques mm au dessous de la surface. Aucune quantité importante de vapeur surchauffée n'a été décelée. La glace se formait au niveau de l'interface et l'augmentation du flux de chaleur s'explique en supposant le passage de l'ébullition en film à l'ébullition muléée. Les mélanges en ébullition moussaient fréquemment et les photographies prises au niveau de l'interface ont montré que de très petites bulles de vapeur se formaient au début de l'expérience.

INSTATIONÄRES SIEDEN VON VERFLÜSSIGTEN GASEN AUF EINER WASSEROBERFLÄCHE—II. GEMISCHE LEICHTER KOHLENWASSERSTOFFE

Zusammenfassung—Gemische aus leichten Kohlenwasserstoffen, verflüssigtes Naturgas, siedeten auf einer Wasseroberfläche, wobei die Verdampfungsrate gemessen wurde. Die Wärmestromdichten waren bedeutend höher, als sie für reines, flüssiges Methan ermittelt wurden, auch wenn dabei die Molanteile von Äthan, Propan und *n*-Butan kleiner 0·01 waren. Wie bei reinem Methan nahm die Verdampfungsrate während eines Versuchs zu, solange sich keine zusammenhängende Eisschicht gebildet hatte. Die Wassertemperatur am Anfang betrug 6 bis 60°C, die Flächenbelastung des LNG (liquefied natural gas) 0·21 bis 0·83 g/cm^2. Bei Gemischen von Methan mit schwereren Kohlenwasserstoffen, bie denen der Molanteil der letzteren Komponenten größer als 2% war, nahm die Oberflächentemperatur schnell die Temperatur der Flüssiggase an, jedoch wurde wenige Millimeter unter der Oberfläche schon eine geringe Temperaturdifferenz festgestellt. Eine starke Überhitzung des Dampfes wurde nicht beobachtet. An der Phasengrenzfläche bildete sich sofort eine Eisschicht, wobei die Wärmestromdichte beim Übergang vom Film- zum Blasensieden zunimmt. Während des Siedens schäumten die Gemische oft auf; fotografische Aufnahmen der Grenzfläche zeigten, daß sich in jedem Versuch frühzeitig sehr kleine Dampfblasen bildeten.

НЕСТАЦИОНАРНОЕ КИПЕНИЕ СЖИЖЕННЫХ КРИОГЕНОВ НА ПОВЕРХНОСТИ ВОДЫ — II. СМЕСИ ЛЕГКИХ УГЛЕВОДОРОДОВ

Аннотация — Смеси легких углеводородов, характерные для сжиженного природного газа, подвергались кипению на поверхности воды, и проводилось измерение скорости их испарения. Значения тепловых потоков были значительно выше полученных для чистого жидкого метана даже в том случае, когда мольные фракции этана, пропана и п-бутана не превышали 0,01. Как и в случае с чистым метаном скорость испарения в течение эксперимента увеличивалась до тех пор, пока не образовывался бесконечный толстый слой льда. Диапазон значений начальной температуры жидкой воды составлял от 6 до 60°С, а количество сжиженного природного газа — от 0,21 до 0,83 г/см^2. Для смесей метана с более тяжелыми углеводородами, когда мольное процентное содержание последних было выше 2%, температура поверхности воды быстро падала до криогенной температуры, однако несколько миллиметров ниже поверхности температура изменялась незначительно. Какого-либо значительного перегрева пара отмечено не было. Лед образовывался быстро на границе раздела, и увеличение теплового потока объяснялось переходом от пленочного кипения к пузырьковому. Отмечалось частое вспенивание кипящих смесей. Фотографии, снятые на границе раздела, показали, что сразу же в начале опыта образовывались очень маленькие пузырьки пара.