1.37 grams (one mol) *p*-aminobenzoic acid in 100 cc. toluene for nine hours under a reflux condenser. The time was increased in this case to reduce the amount of a by-product melting at 171–3°. The chief product, which crystallized out on cooling, consisted of pale yellow prisms and could be recrystallized from toluene or water. The pure substance melts at 233°–4°.

Calculated for $C_{15}H_{11}O_4N$: C, 66.90; H, 4.08
Found: C, 67.14; H, 4.64

If in the preparation of this compound the boiling was interrupted after two or three hours, a small amount of a substance melting at 171–3° could be readily isolated. The amount could be greatly increased by employing two molecules of the acid for one of the aldehyde, but no satisfactory analytical figures could be obtained for a product containing two acid residues, in spite of many analyses.

3,4-Methyleneoxybenzalethyl-p-aminobenzoate, $CH_2 : O_2 : C_6H_3CH : NC_6H_4\text{-}CO_2C_2H_5$, is prepared by boiling 1.50 grams piperonal and 1.65 grams ethyl-*p*-aminobenzoate in 10 cc. benzene for six hours. The product crystallizes poorly from benzene so the latter is evaporated off. Out of ligroin the compound crystallizes readily in long, pale yellow, glistening needles which melt at 109° (cor.).

Calculated for $C_{15}H_{15}O_3N$: C, 68.66; H, 5.08
Found: C, 68.96; H, 5.04

3,4-Methyleneoxybenzal-p-anisidine, $CH_2 : O_2 : C_6H_3CH : NC_6H_4OCH_3$. The condensation of piperonal with *p*-anisidine is carried out exactly as that of vanillin with *p*-anisidine. The product crystallizes well from ligroin or benzene. The crystals are very pale yellow needles, which separate in feathery groups. Melting point, 117.5° (cor.).

The experimental work here described was carried out by Mr. L. E. Stacy, Jr., and Mr. L. B. Rhodes and I wish to thank them for their careful work.

CHAPEL HILL, N. C.

[CONTRIBUTION FROM THE EXPERIMENTAL STATION OF THE E. I. DUPONT DENEMOURS POWDER CO., WILMINGTON, DELAWARE.]

THE FREEZING OF NITROGLYCERINE.

THE HEAT OF TRANSFORMATION OF THE NITROGLYCERINE ISOMERIDES.

BY HAROLD HIBBERT AND G. PRESCOTT FULLER.

Received May 12, 1913.

Introduction.

In a previous communication on "The Preparation, Crystallin Structure and Physical Properties of the Two Forms of Solid Nitroglycerine,"[1] the preparation and some of the physical properties of the two isomeric

---

[1] *International Congress of Applied Chemistry, N. Y.* (1912), *Section III-b*, page 37.

forms of solid nitroglycerine were described. In the course of that work it was found (p. 46) that if a quantity of the *labil* solid isomeride was inoculated with a crystal of the stable variety and stirred vigorously, a certain rise in temperature occurred. When liquid nitroglycerine was inoculated with the *stable* isomeride under the same conditions, the evolution of heat was found to be of approximately the same order of magnitude as in the former case, and the conclusion was therefore drawn that in the two processes:

1. Liquid Phase ⟶ Labil Solid Phase
2. Labil Solid Phase ⟶ Stable Solid Phase

the heat tone of the first was relatively small in comparison with that of the second. That this was indeed the case was also indicated by the great readiness with which the labil form could be transformed into the stable isomeride. As the above-mentioned experiments were only of a qualitative nature, it was thought desirable to make accurate calorimetric determinations of the heat changes involved in the isomerization. The only previous work on this subject is that of Nauckhoff,[1] who determined the evolution of heat taking place during the crystallization of liquid nitroglycerine into the stable solid form, the mean value found being 23.1 calories per gram at +1.5°. His experiments were performed with the aid of a mercury calorimeter and the value found is of doubtful accuracy on account of the experimental difficulties involved in the use of such an apparatus, especially in a case where the evolution of heat continues to take place over a period of from 30 to 60 minutes. Nauckhoff himself apparently did not place great reliance upon his results for he says in this connection,[2] "Es wäre sehr zu wünschen dass diese Bestimmungen an einem wissenschaftlichen Institut mit groesseren Hilfsmitteln, als die mir zu Gebote stehenden, wiederholt würden. Ich beabsichtige, sobald sich eine Gelegenheit dazu bietet, die latente Schmelzwärme..........des Nitroglycerins unter Zuhilfenahme des Bunsenschen Eiskalorimeters von Neuem zu bestimmen." The nitroglycerine used for his work was also somewhat impure as its freezing point was 12.3° C., whereas pure nitroglycerine freezes (as the stable form) at 13.0° C. No experiments with the labil form have hitherto been carried out, for the simple reason that, prior to the investigation mentioned above, the conditions for its preparation had apparently not been definitly established. Nauckhoff indeed had entirely failed[3] in his effort to repeat Kast's work[4] on the preparation of the labil isomeride.

Acting on Nauckhoff's suggestion it was decided to employ the Bunsen calorimetric method, since the latter undoubtedly possesses many ad-

---

[1] *Z. angew. Chem.*, **1905**, 17.

[2] *Loc. cit.*, p. 19.

[3] *Z. ges. Schiess-Sprengstoffwesen*, **6**, 124 (1911).

[4] *Ibid.*, **1**, 225 (1906).

vantages over that of the mercury calorimeter, chief among which is the fact that the heat given out in the isomerization process is immediately absorbed by the ice in melting and the loss of heat by radiation, etc., is therefore negligible. The time occupied in the conversion of the liquid into the labil solid form for instance is about two hours, and the loss of heat by radiation during this time, in a system intended to be adiabatic, might amount to quite a large proportion of the total heat evolved. Such is not the case, however, in an isothermal system.

As indicated later in detail, two sets of experiments were carried out, the results obtained in the second set (tabulated below) being the more accurate on account of the experimental conditions being under much better control.

Nature of the change taking place.	Heat evolved in gram calories.	Heat evolved in molar calories.
1. Liquid phase to labil solid phase	5.2	1180.4
2. Labil solid phase to stable solid phase	28.0	6356.0
3. Liquid phase to stable solid phase	33.2	7536.4

Our value of 33.2 calories per gram for the total heat change of:

$$\text{Liquid Phase} \longrightarrow \text{Stable Solid Phase}$$

at 0° C., thus differs from that of Nauckhoff (measured at +1.5° C.) by about 43%; the difference in temperature corresponds, however, to only 0.47 calories. The heat change of 5.2 calories per gram, involved in the transformation:

$$\text{Liquid} \longrightarrow \text{Labil Solid}$$

represents only 15% of the total heat change involved in the final formation of the stable solid, thus confirming the deductions made in our previous investigation.

It is of some interest to compare the above values with two other physical constants of nitroglycerine, *viz.*, its heat of formation (98,900 calories) and the heat of combustion (432,101 calories at constant volume). The latent heat of fusion of the stable isomer amounts to 7.6% of the former, and 1.7% of the latter value and thus represents no insignificant thermal change when viewed from this standpoint, and especially from the fact that the nitroglycerine molecule itself must be regarded as representing a very sensitive, if not relatively unstable, molecular aggregation.

In the course of the former investigation of the nitroglycerine isomerides it was found[1] that if the labil solid was inoculated with a crystal of the stable form and the mixture thoroughly incorporated in a mortar, the whole mass was converted almost instantly into the stable variety. It seemed of interest therefore to determin the velocity of transformation when the crystals were merely in contact, all stirring or mixing being

---

[1] *Loc. cit.*, p. 47.

avoided. It was found that the isomerization was complete in from 20 to 40 minutes, the difference in time being doubtless due to the element of chance in the surface of each crystal being in more or less intimate contact with the surface of the other form, and thus determining the velocity with which the transformation proceeds.

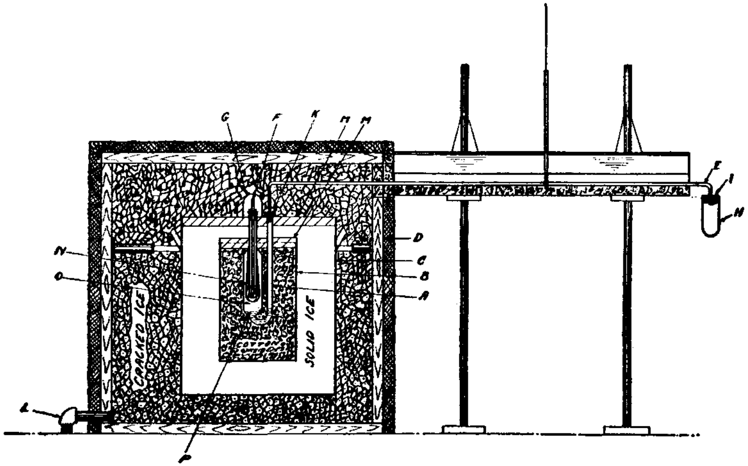
Experimental Part.

I. Manipulation.

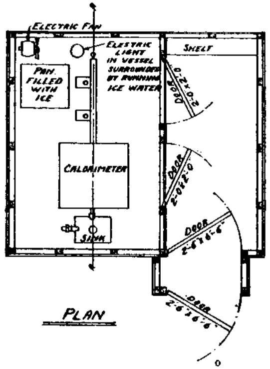
The theory of the Bunsen ice-calorimeter is so familiar to everyone that no detailed description is necessary. The most important precautions to be taken in the use of this apparatus are (*a*) effective insulation to prevent any loss of heat by radiation, (*b*) the careful exclusion of all air from the water in the calorimeter and (*c*) the use of pure ice in packing. The general arrangement of the apparatus is indicated on the accompanying sketch.

Preparation of the Ice-Calorimeter.—The glass calorimeter was cleaned with both chromic acid and aqua regia, then allowed to soak in water for a week, the water being changed every day. An amount of pure, freshly cleaned, and redistilled mercury, sufficient to form a 3 cm. layer on the bottom of the calorimeter, was poured in through the side arm, and the top then completely filled with freshly distilled water. By boiling out under reduced pressure (the tube being immersed in water heated to about 60°), all the air in the water was completely expelled. The water in the side arm was then pipetted out, the tube thoroughly dried out and then filled with pure mercury, the presence of air bubbles in the latter being carefully avoided. It was next necessary to form a cylinder of ice about the inner tube of the calorimeter, and this was found to be most readily brought about by cooling the calorimeter in ice and then forming the ice cylinder by introducing cold, dry ether at about —10° C. into the inner tube and blowing a rapid current of dry air through it. When freezing first commenced the whole interior of the calorimeter was found to be filled with plates of ice, but by stroking with the hand these could be melted, leaving only a thin film of ice around the inner tube. Cold, dry ether was then introduced a little at a time, and by blowing dry air through it, a perfectly solid and symmetrical cylinder of ice could be readily formed, the ice coating being made largest at the bottom. As the temperature of the calorimeter and its contents was now below 0° it was packed in ice and allowed to stand for 24 hours before being transferred to the insulating boxes. Just prior to placing it in the box, however, the side arm was connected with the vacuum pump and the tube tapped vigorously to expel any bubbles of air that might have been included by the mercury column.

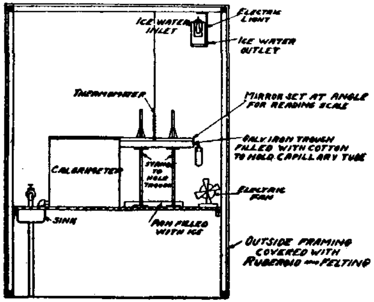
Transformation of Liquid Phase into Labil Solid Phase.—After standing for several days in order to allow the system to come to a state of temperature equilibrium, a weighed amount of previously cooled liquid nitroglycer-



SECTION A-A



PLAN

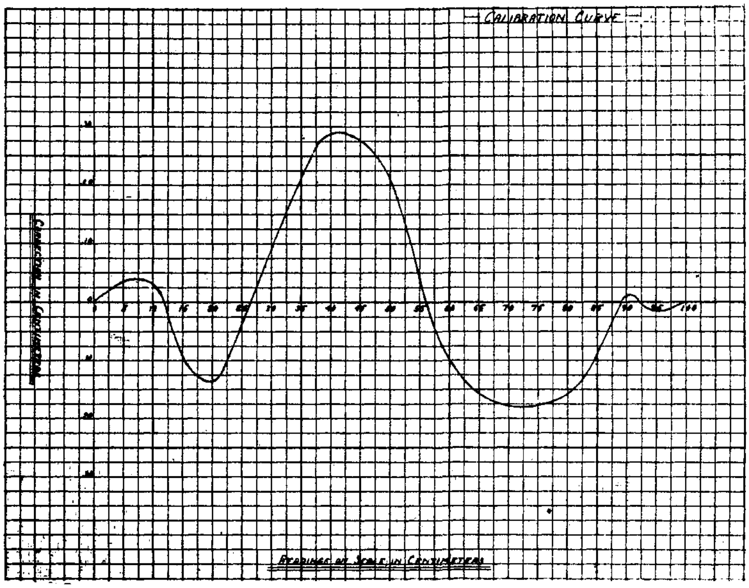


SECTIONAL ELEVATION

APPARATUS FOR DETERMINATION
OF
HEAT OF TRANSFORMATION
OF
NITROGLYCERINE ISOMERIDES

SCALE: ⅜" = 1'-0"
1-15-19

ine was introduced into the inner tube of the calorimeter. In order to avoid the loss of any of the liquid, the nitroglycerine was weighed in a pipet, then a portion introduced and the weight of the amount used determined by difference. Measurements of the "creep" of the mercury thread were now made every 30 minutes and after the motion had become very small, every 15 minutes. As soon as this was found to be either zero, or else very small, but constant, the stirrer (which consisted of a piece of glass rod on the lower end of which had been attached a series of small glass projections so as to assist in the more rapid impregnation of the total mass after inoculation) was removed, dipped in a mass of freshly frozen crystals of the labil form of solid nitroglycerine cooled to 0° C. (whereby extremely minute amounts of the crystals were retained by the fine glass points of the stirrer), then at once returned to the calorimeter and the sides of the tube under the surface of the liquid scratched for a short time. Under these conditions it was found that crystallization commenced in a few minutes and was usually complete in the course of about two hours. (As a stirrer of smooth glass did not give satisfactory results the above type was devised and found to work very efficiently in promoting rapid crystallization.) During this period, observations of the position of the mercury thread were made every five minutes for the first half hour, every ten minutes for the next half hour, and after that every fifteen minutes for at least three hours, in order to determin the magnitude of the "creep" after the experiment. It is to be noted that the weight of nitroglycerine introduced in the process of in-



oculation was altogether a negligible quantity in comparison with the weight of nitroglycerine originally taken; further, the form of the accompanying curve indicates that no appreciable error was introduced by any minor change in temperature of the glass stirrer during this operation.

Transformation of the Labil Solid into the Stable Solid Phase.—When the motion had again become constant a very small crystal of the stable form of nitroglycerine was introduced and the mass stirred vigorously for one-half minute in order to bring about a rapid inoculation of the solid labil isomeride with the solid stable form. In this second case crystallization generally commenced in a very few minutes and was usually practically complete in 30 minutes, the "creep" being observed as in the previous case for a period of two hours or until it had become constant.

The liquid nitroglycerine employed in this investigation was prepared from "dynamite glycerine" and an acid of the following composition:

H_2SO_4	61.35%
HNO_3	32.16%
$H(NO)SO_4$	1.46%
H_2O	5.03%
	100.00%

After careful purification and drying it was recrystallized three times through the labil form, half of the product being rejected in each portion, this procedure having been found, as indicated in the previous paper,[1] to be the most advantageous for the isolation of a product of a high degree of purity.

Statement of Experimental Difficulties Involved.—In the course of this investigation many experimental difficulties were encounterd, the solution of some of which consumed a large amount of time. The most important of these was the fact that for a long time it was found impossible to prevent a continuous outward motion of the mercury in the capillary tube. This was an indication of a freezing process going on in the calorimeter itself, and in fact when the glass calorimeter was examined after a determination, it was almost invariably found to contain more ice than when originally set up. As a matter of fact, the formation of this excess of ice sometimes persisted so long that the calorimeter was cracked by the resulting expansion of the ice. The cause of this phenomenon was finally resolved into two factors: In the first place when the ice cylinder in the glass calorimeter was formed, the temperature of the whole was brought below 0° and being inserted in this condition into the system of insulating boxes, no interchange of heat could take place with the exterior and therefore in order to come to equilibrium more ice was frozen until the temperature had come up to 0°. A calculation showed that if the temperature

---

[1] *Loc. cit.*; p. 43.

of the calorimeter and its contents were only —1°, this would cause a total motion of approximately 1000 cm. before the temperature rose to 0°. This effect was subsequently avoided by packing the calorimeter in pure ice, after forming the ice cylinder, and allowing it to stand at 0° for 24 hours. The temperature was thus brought up to practically 0° before introducing it into the insulating system. Then again in the formation of the distilled water ice-cake in the inner box (C) the temperature of this was brought down to —5° to —8°, while in the brine tank, and if placed in the insultating system in this condition, several weeks were found to elapse before this temperature rose to 0° due to the high efficiency of the insulation. This effect was avoided to a great extent by allowing the box containing the freshly frozen cake to stand in ice water for a number of hours before putting it in place.

The second factor was concerned with the ice used in packing the outer box. For several months ordinary commercial ice was used for this purpose. However, as the outward motion of the mercury thread was not prevented by overcoming the first factor mentioned above, it was suspected that this commercial ice might contain a trace of some saline impurity, thus lowering its temperature slightly below 0°. Calculations based upon an actual determination of the amount of mercury forced out from the calorimeter during a period of five weeks showed that 32.8 grams of water had been frozen, equivalent to an absorption of 2624 calories. Assuming the mass of the packing ice as approximately 100 kilograms, the total motion of the mercury thread would be accounted for if the temperature of the packing ice were but 0.025° below zero. The use of commercial ice was then given up altogether and distilled water ice substituted. A marked change in the behavior of the apparatus was immediately observed and it was found that the attainment of equilibrium under the new conditions required only a relatively short time.

Another difficulty encountered, especially during the early portion of the work, was the fact that the motion of the mercury was not constant but fluctuated badly at times. This was probably due to several causes. In the first place, the capillary tube had such an exceedingly small bore that the presence of the smallest particle of dust, scum on the mercury, or even moisture, was found to be sufficient to stop the motion temporarily, until the accumulated stress set up by the continued freezing of ice inside the tube was sufficient to overcome the friction, whereupon a very sudden and rapid motion would occur. Another factor of only minor importance was the variation in barometric pressure. Given an initial condition of equilibrium, an increase in external pressure should cause a very slight outward motion due to the change in volume of the glass calorimeter. However, as an initial condition of outward motion usually obtained this effect would only manifest itself in an acceleration of the movement. This effect would be counterbalanced by the added resistance to the motion of the mercury thread, due to the increased pressure. The sum total of these two factors was assumed to be negligible in the calculations. A third factor having to do with the observed variations was the temperature of the room. When the mercury left the calorimeter it was at 0°, whereas the room possessed a mean temperature of 12°. Accordingly, the mercury commenced to expand as soon as it entered the capillary, and the further out the meniscus the greater the expansion and the more rapid the observed motion.

In the actual determinations a difficulty of a different type was sometimes encountered, *viz.*, a spontaneous crystallization of the nitroglycerine after its introduction into the calorimeter. In one experiment in particular pure liquid nitroglycerine was placed in the tube, and then allowed to stand with the tube tightly stoppered, until temperature equilibrium had been attained. As no motion of the mercury was caused by seeding with a crystal of the solid isomeride the tube was examined and the nitroglycerine found to have been completely converted into its stable solid form during this time of standing. This phenomenon was also observed during the previous investigation of the solid isomerides, the crystallization sometimes occurring from no apparent cause.[1] The cause of this spontaneously occurring crystallization could not be positively determined, but it was probably due to inoculation of the liquid by solid nuclei in the air during the time of weighing. The only other possibility of infection would be in the presence of some of the solid in the tube itself, but as the latter had been washed out repeatedly with warm ether after the previous experiments, it does not seem possible that any could have remained.

With regard to the capillary tube employed, this had previously been carefully graduated so that from the data we were able to compute the weight of ice which would have to be melted in order to cause a diminution of volume sufficient to move the mercury thread a distance of one cm. Knowing the latent heat of fusion of ice we could then calculate the number of calories of heat corresponding to a motion of 1 cm.

In every experiment, just prior to the assembling of the apparatus, the capillary tube was cleaned out thoroughly by sucking through it in succession, (1) warm aqua regia, (2) pure water, (3) dry alcohol, (4) dry ether, and finally (5) dry air, filtered through cotton-wool, for at least one-half hour.

The results shown below have been corrected for temperature, "creep" and inequalities of the scale.

II.—Calibration Data.

The volume of the capillary tube was determined by weighing the thread of mercury contained in it.

---

[1] *Loc. cit.*, p. 17.

READINGS OF MERCURY THREAD.

Length. Cm.	Weight. Gm.	Temp.	Density of mercury.	Total volume. Cc.	V_s. Cc.
99.9362	0.62830	30.5°	13.5206	0.04647	0.0004649
99.9740	0.63025	31.5°	13.5181	0.04662	0.0004663
99.8460	0.63038	32.3°	13.5162	0.04664	0.0004671

Mean value V_s = 0.0004661

Calculation of Value of 1 cm. of Scale in Calories.

V_s = 0.0004661 cc. = Volume of each cm. of tube
a = 0.0001818 = Coefficient of expansion of mercury
d = 0.91674 = Density of ice at 0°
e = 0.99987 = Density of water at 0°
l = 80.025 = Latent heat of fusion of ice
Volume of 1 gram of ice at 0° = l/d = 1.09082 cc.
Volume of 1 gram of water at 0° = l/e = 1.00013 cc.
Weight of ice melted to move meniscus 1 cm. = $V_s/l/d - l/e$ = 0.005139 gram.

Therefore, the heat evolution necessary to move the meniscus 1 cm. is equal to 80.025 × 0.005139 = 0.4113 calories.

For the above data we are indebted to Dr. R. W. Duncan of the Physics Department of the University of Pennsylvania who loaned us the capillary tube in question, and to whom we wish to express our hearty thanks for his kindness in this connection. We satisfied ourselves as to the accuracy of the figures prior to the commencement of the investigation.

III.—Experimental Data on Heat of Transformation.

Exp. I (a) Transformation of Liquid Phase into Labil Solid Phase.

Weight of liquid nitroglycerine.......... 0.8294 gram
Creep before the experiment = —0.7 cm. in 135 minutes
Total motion.......... 12.5 cm.
Time.......... 120 mins.
Creep after the experiment = —6.0 cm. in 180 minutes
Average creep = $^1/_2$ (—0.7/135 — 6.0/180) = —0.019 cm. per minute
Correction for creep = (120) (—0.019) = —2.28 cm.
Temperature of room.......... 12.5°
Correction for temperature = —(0.000182) (12.5) (12.5) = —0.03 cm.
Correction for inequality of scale = +0.025 — 0.105 = —0.080 cm.
Corrected motion = 12.5 — 2.28 — 0.03 — 0.08 = 10.1 cm.
1 cm. ≐ 0.4113 calories ∴ 10.1 cm. = (10.1) (0.4113) = 4.15 calories
Latent heat of *Liquid Phase* ⟶ *Labil Solid Phase* = 4.15/0.8294 = *5.0* calories

Exp. I (b) Labil Solid Phase ⟶ Stable Solid Phase.

Weight of liquid nitroglycerine.......... 0.8294 gram
Creep before the experiment = —6.0 cm. in 180 minutes
Total motion.......... 60.0 cm.
Time.......... 90 mins.
Creep after the experiment = —5.5 cm. in 60 minutes
Average creep = $^1/_2$ (—6.0/180 — 5.5/60) = —0.062 cm. per minute
Correction for creep = (90) (—0.062) = —5.58 cm.
Temperature of room.......... 12.5°

Correction for temperature = —(0.000182) (12.5) (60) = —0.14 cm.

Correction for inequality of scale = +0.005 — 0.055 = —0.050 cm.

Corrected motion = 60.0 — 5.58 — 0.14 — 0.05 = 54.2 cm.

1 cm. $\doteq$ 0.4113 calories ∴ 54.2 cm. = (54.2) (0.4113) = 22.29 calories

Latent heat of *Labil Solid Phase* → *Stable Solid Phase* =
22.29/0.8294 = *26.9* calories

Latent heat of *Liquid Phase* → *Stable Solid Phase* =
5.0 + 26.9 = *31.9* calories

Exp. II (a) Liquid Phase → *Labil Solid Phase.*

Weight of liquid nitroglycerine.............................. 0.5536 gram

Creep before the experiment = —0.2 cm. in 40 minutes

Total motion.. 7.3 cm.

Time.. 125 mins.

Creep after the experiment = 0.0 cm. in 180 minutes

Average creep = $^1/_2$ (—0.2/40 + 0/180) = —0.002 cm. per minute

Correction for creep = (125) (—0.002) = —0.25 cm.

Temperature of room...................................... 11.0°

Correction for temperature = —(0.000182) (11.0) (7.3) = —0.01 cm.

Correction for inequality of scale = +0.038 — 0.060 = —0.022 cm.

Corrected motion = 7.3 — 0.25 — 0.01 — 0.02 = 7.0 cm.

1 cm. $\doteq$ 0.4113 calories ∴ 7.0 cm. = (7.0) (0.4113) = 2.88 calories

Latent Heat of *Liquid Phase* → *Labil Solid Phase* =
2.88/0.5536 = *5.2* calories

Exp. II (b) Labil Solid Phase → *Stable Solid Phase.*

Weight of nitroglycerine.................................. 0.5536 gram

Creep before the experiment = 0.0 cm. in 180 minutes

Total motion.. 37.7 cm.

Time.. 33 mins.

Creep after the experiment = —0.4 cm. in 180 minutes

Average creep = $^1/_2$ (0/180 — 0.4/180) = —0.001 cm. per minute

Correction for creep = (33) (—0.001) = —0.03 cm.

Temperature of room...................................... 11.0°

Correction for temperature = —(0.000182) (11.0) (37.7) = —0.08 cm.

Correction for inequality of scale = —0.060 + 0.175 = +0.11 cm.

Corrected motion = 37.7 — 0.03 — 0.08 + 0.11 = 37.7 cm.

1 cm. $\doteq$ 0.4113 calories ∴ 37.7 cm. = (37.7) (0.4113) = 15.50 calories

Latent Heat of *Labil Solid Phase* → *Stable Solid Phase* =
15.50/0.5536 = *28.0* calories

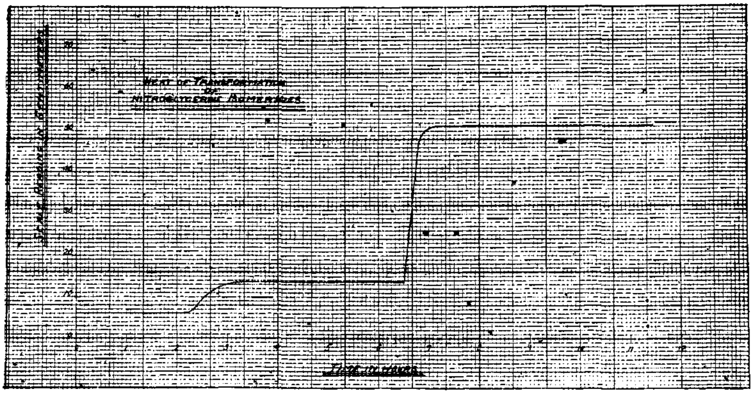
Latent Heat of *Liquid Phase* → *Stable Solid Phase* =
5.2 + 28.0 = *33.2* calories

A graphic representation of the heat changes (Expt. 2) is shown on the accompanying curve.

IV. Experiments on the Velocity of Isomerization.

Several sets of experiments were performed with the object of determining the velocity of isomerization under various conditions.

A large crystal of the labile form of nitroglycerine was placed in each of several tubes and the latter stoppered and cooled in ice. A minute crystal of the stable variety was dropped into each and then the melting point of the contents of each tube determined after intervals of 5, 10, 15, etc., minutes. In no case had the labile form undergone any change into the stable in a period of less than 20 minutes, but in from 25 to 35 minutes, the change was complete in practically every case.



When crystals of the labile form were inoculated with the stable variety at 0° and the mixture stirred vigorously with a *pointed stirrer*, the transformation completed itself in from 3 to 6 minutes.

Liquid nitroglycerine cooled to 0°, inoculated with the labile form and stirred with a *pointed stirrer* for from two to four minutes, usually completely solidified in from 45 to 60 minutes.

Summary.

1. The heat of transformation of one gram liquid nitroglycerine into its labil solid isomeride has been found to be 5.2 calories.

2. The heat of transformation of one gram labil solid nitroglycerine into the stable solid isomeride has been found to be 28.0 calories.

3. The heat of transformation of one gram liquid nitroglycerine into its stable solid form is therefore 33.2 calories.

4. It has been determined that on mere contact with a crystal of the stable variety, the labil solid form can be completely converted into its isomeride in approximately 30 minutes (at 0°). By thoroughly incorporating the two by means of a pointed glass stirrer, the change can be made to take place completely in some three to four minutes.

The above investigation was carried out at the Experimental Station of the E. I. DuPont deNemours Powder Company, Wilmington, Delaware, and the authors wish to thank the Director and the officials of the Company for their permission to publish the same.