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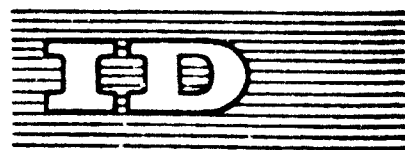
United Nations Industrial Development Organization

Second Interregional Fertilizer Symposium

Kiev, USSR, 21 September - 1 October 1971
New Delhi, India, 2 - 13 October 1971

Agenda item III/le/ii

SUMMARY

TECHNIP MAVROVIC HEAT RECYCLE UREA PROCESS[1]

by

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The "Heat Recycle" urea synthesis process is characterized by three basic improvements attained over the conventional urea synthesis processes:

1) Reduction in plant investment cost.

2) Reduction in utility consumption.

3) Increase in process and mechanical reliability of the plant.

The reduction in plant investment cost is attained in the H.R. Urea Process by improving the efficiency and the conversion in the urea synthesis reactor. As a consequence the amount of the carbamate recycle and the size of the equipment handling the carbamate recycle are drastically reduced.

The reduction in utility consumption is attained by internally recovering the heat of reaction of NH_3 and CO_2 to carbamate. In conventional urea synthesis processes such heat of reaction is irreversibly rejected to the cooling water, whereas in the H.R. urea process the heat of reaction of NH_3 and CO_2 to carbamate is transfered to the reactor

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feed streams. Consequently the urea synthesis reactor becomes strongly exothermic, in contrast to the adiabatic reactor of a conventional urea process. In the H.R. urea process the excess heat added to the reactor is removed from it by the conventional method of producing steam in the reactor coil. In the H.R. urea process such steam is produced at a pressure level sufficient to be utilized to decompose the carbamate contained in the reactor effluent to NH_3 and CO_2 gas, which is separated from the urea product solution. About 85% of the heat released by reacting the NH_3 and CO_2 gas in the recovery and absorption section is recovered in exchange with the reactor feed streams and recycled to the reactor. The exothermic reactor medium pressure steam is produced in a sufficient amount to supply about 55% of the steam required in the cabamate decomposition stage.

The process and the mechanical reliability of the plant is attained by simplifying the process and the equipment design.

The process stability is attained by controlling the synthesis loop particularly with respect to carbamate recycle solution concentration, thus completely eliminating the possibility of solution crystallization and consequent mechanical failure of the equipment. The process stabilization of the urea synthesis loop is attained in the H.R. urea process by a novel method of reducing the content of water vapour in the NH_3 and CO_2 gas produced in the carbamate decomposers and of maintaining it within a predetermined range. Such water vapour content in the carbamate decomposer gas is in turn automatically controlled by a newly developed continuous concentration analyzer, installed on the carbamate recycle solution reactor feed line containing the solution obtained from condensation of the carbamate decomposer NH_3 and CO_2 gas.

The solution concentration analyzer is directly calibrated in terms of crystallization temperature of the carbamate recycle solution, thus facilitating the task of the urea plant operator.

The equipment design is, in general, simplified in design and mainly reduced to simple and conventional type shell and tube heat exchangers. A very limited amount of equipment with moving or rotating parts is used in addition to the high pressure pumps for liquide NH_3 and carbamate solution, and the CO_2 gas compressor.

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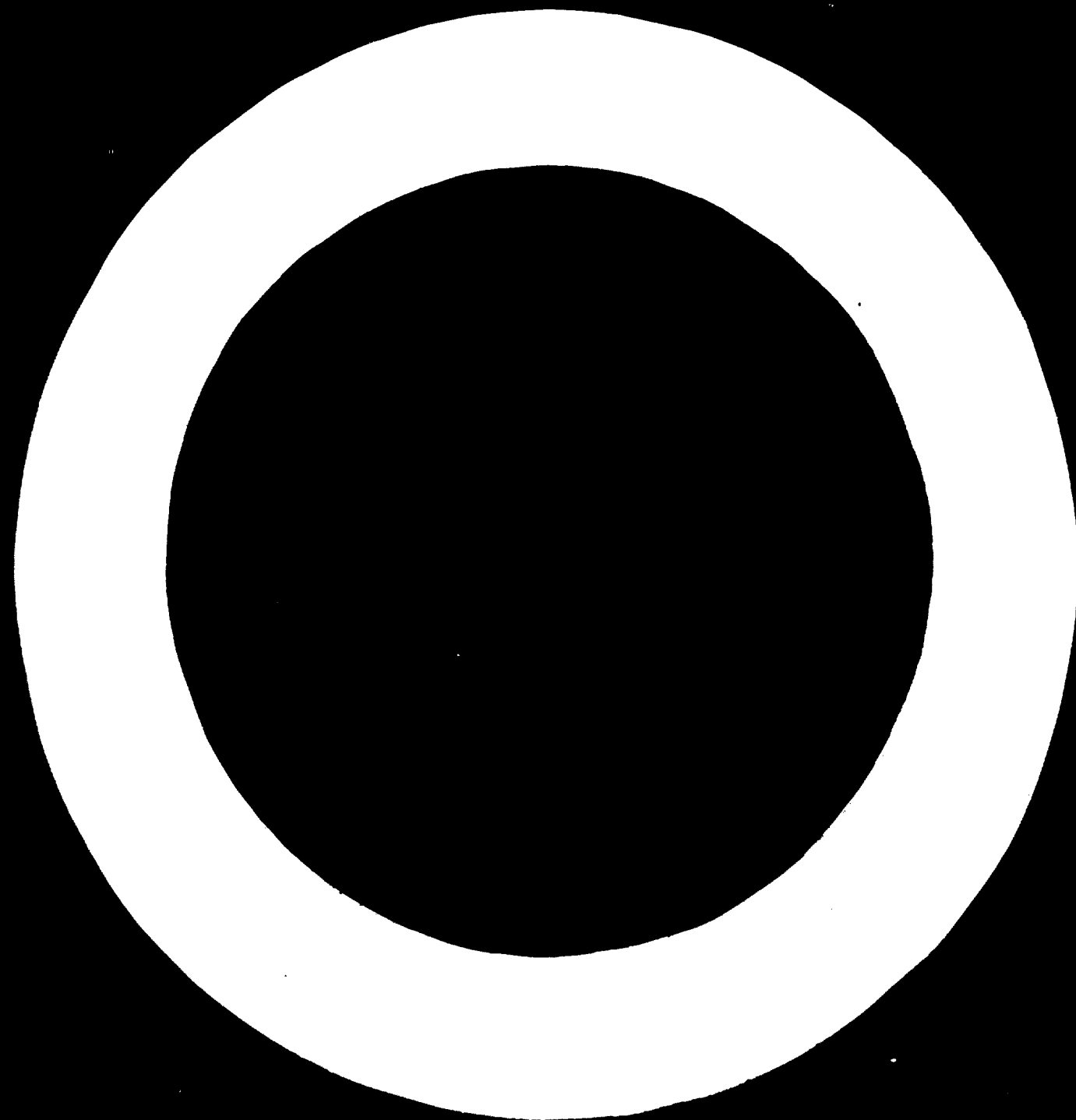
TECHNIP MAVROVIC HEAT RECYCLE UREA PROCESS[1]

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1 - GENERAL

The urea marketing conditions troughout the world have been growing increasingly competitive during the past several years, and consequently the urea producers' demands on the urea technology for a reduction in urea production cost have been becoming stronger.

As a result, the new improvements have been aimed at one or more of the main factors affecting the urea production cost : reduction in plant investment cost, reduction in utility consumption, improvement in process reliability and plant reliability.

It became evident that a substantial reduction in plant investment cost and in utility consumption could be attained by increasing the efficiency and the conversion per pass in the urea synthesis reactor, and by recovering the heat of formation of ammonium carbamate from gaseous NH_3 and CO_2. At increased reactor efficiency the highest possible conversion per pass in the reactor is attained at a given ammonia to carbon dioxide feed mol ratio and at given reactor operating pressure and temperature conditions. At higher reactor conversion per pass the equipment handling the recycled NH_3 and CO_2 is drastically reduced in size and in addition the energy required to recover the unconverted reactants NH_3 and CO_2 from the urea solution product is reduced. By internally recovering the heat of reaction of carbamate formation the heat input into the battery limits urea plant and the cooling water consumption are proportionally decreased.

Furthermore, by adopting long and well proven conventional process conditions and equipment with basic improvements in design and in method of process control, the plant on stream period is extended considerably, well beyond the usually attained period of 320-330 days per year.

The "Heat Recycle Urea Process" is the result of the combination of practically all of the above mentioned improvements.

2 - PROCESS DESCRIPTION

The "Heat recycle" process is a liquid carbamate recycle urea process with excess NH_3 gas recovery at the standard 15-25 ATM. level of first stage carbamate decomposition, followed by a low pressure urea product solution degassing and NH_3 - CO_2 recovery stage.

The degassed 76 wt % urea product aqueous solution is concentrated to about 85 wt % solution, to be further processed in either a crystal melting system or an evaporator system to produce pure molten urea which is spray-cooled into solid urea prills.

Urea reactor

The urea synthesis reactor operates at a temperature of 190 °C and at a pressure of 225 atmospheres, at an overall NH_3 to CO_2 molar ratio of about 4,2 to one and at a H_2O to CO_2 molar ratio of about 0,6 to one. The overall conversion per pass of total CO_2 to urea is about 72 %. Atmospheric air is compressed to reactor pressure, mixed with the liquid NH_3 reactor feed stream and introduced into the urea synthesis reactor for the purpose of passivating the 316 L SS reactor liner and completely eliminating corrosion of the high pressure equipment in contact with the urea synthesis mixture. The amount of reactor air feed is maintained at a relatively low value, equivalent to about 0,7 parts of CO_2 per 1,000 parts of fresh gaseous CO_2 make up to reactor, for the specific purpose of eliminating the ill effects of the presence of an excessive amount of inerts and oxygen in the urea synthesis reactor. An excessive amount of inerts is detrimental to the conversion, and an excessive amount of oxygen is detrimental to the product quality due to the impurities which can form in the presence of excess O_2 in the reactor mixture. This fact is especially important in the production of technical grade urea containing 0,5 - 0,6 wt % by-product biuret, obtained by direct evaporation of the 75 wt % aqueous urea product solution and by prilling the pure urea melt thus obtained.

It is well known to the urea operator from practical experience that the performance of the urea plant depends upon the performance of the reactor : at low conversion the carbamate load on the decomposers and absorbers increases as along with the plant utility consumption ; at relatively high NH_3 to CO_2 reactor feed mol ratio the reactor conversion can be increased up to a certain level, but usually at the expense of overloading the equipment with excess ammonia gas and increasing the ammonia losses.

The relationships between the NH_3 and H_2O to CO2 reactor feed mol ratios and the conversion of CO2 to urea at a certain pressure and temperature is expressed by the equation for the equilibrium constant "K" as defined by Frejacques :

$$K = \frac{x\,(b + x)\,(1 + a + b - x)}{(1 - x)\,(a - 2x)^2} \qquad (A)$$

where :   a = NH_3 to CO_2 reactor feed mol ratio

b = H_2O to CO2 reactor feed mol ratio

x = fraction of CO_2 converted to urea, in reactor effluent.

According to formule (A) excess NH_3 increases the conversion x and excess H_2O decreases it.

Nomograph # I here attached, originally calculated and designed by I. MAVROVIC in 1963 is a mathematically exact presentation of the above equation (A).

The original Frejacques determinations of the values for "K" in relation to temperature is directly correlated on the nomograph # I with the left hand temperature scale n° 1 and the "K" scale n° 3.

With the aid of the graph, it is possible to determine the equilibrium conversion "x" for given NH3 and H_2O to CO2 feed mol ratios and for a given operating temperature.

* Note :   Published in "Hydrocarbon Processing" Feb. 66.

For some time it was thought that the values for the equilibrium constant "K" in relation to temperature determined by Frejacques in his experiments and consequently the values for the conversion "x" truly represent ultimate equilibrium values.

MAVROVIC, however, not long ago proved in practice that continuous reactors, designed according to specific and characteristic criteria, yield better results than those obtained from graph # I. The results of these findings were recently published in the April 1971 edition of Hydrocarbon Processing. In the revised nomograph # 2 reported in this publication the left hand temperature scale n°1 was revised.

By taking the actual field data for the NH_3 to CO_2 mol ratio = a, for the H_2O to CO_2 mol ratio = b and conversion = x, and by inserting these empirically obtained values in the right hand side of the above mentioned equation (A), a certain value "K" is calculated. By correlating these newly obtained "K" values with the operating temperatures in the reactor, MAVROVIC found that the above "K" values back calculated from actual field data were relatively higher than those determined by Frejacques and reported on nomograph # I.

On these bases nomograph # 2 was prepared and MAVROVIC's findings incorporated in the revised temperature scale n°1 on the left hand side of the nomograph. This revised temperature scale directly correlates the empirical values for "K" actually obtained from field data and the reactor operating temperature. Such empirical values for "K" are called "efficiency" values for convenience.

In order to illustrate what was said above, a practical example is given as follows : according to nomograph # I based on the "K" values determined by Frejacques, for the reactor temperature of 190 °C a "K" value of 1.45 is determined. At this "K" value, at an NH_3 to CO_2 feed mol ratio of 4.5 to one and at a H_2O to CO_2 feed mol ratio of 0.35 to one it is possible to attain a maximum conversion of 72 %.

On the contrary, MAVROVIC proved in practice that a reactor properly designed, operating at the same temperature of 190 °C, yields a conversion of 72 % at a relatively much lower NH_3 to CO_2 mol or ratio of 4.0 to one and a relatively higher H_2O to CO_2 molar ratio of 0.5 to one. The back calculated value for "K", or "efficiency" as defined above, in this case is equal to 2.38, compared to the "K" value of 1.43 originally predicted on the basis of Frejacques determinations performed in batch autoclaves.

It is a well known fact that in conventional carbamate recycle processes a conversion of 64 - 65 % instead of 72 % is usually attained in practice at an NH_3 to CO_2 mol ratio of 4.0 to one and a H_2O to CO_2 mol ratio of 0.5 to one.

It is also well known that it would be practically impossible to attain the combination of an NH_3 to CO_2 mol ratio of 4.5 to one and a H_2O to CO_2 mol ratio of 0.32 to one in a conventional total recycle urea plant. Such a relatively large amount of excess ammonia would cause too much water vapor to be stripped from the urea product solution, with the consequence of obtaining a too dilute carbamate recycle solution reactor feed. Normally the ultimate H_2O to CO_2 mol ratio at equilibrium in a conventional urea synthesis process operated at a 4.5 to one NH_3 to CO_2 molar ratio is in the range of 0.8 - 0.9 to one.

For the reasons explained above the advantages of the M.M. urea process are obvious. Moreover, as it will be explained further below, the carbamate decomposers and associated equipment are provided with a novel design which allows the use of relatively high NH_3 to CO_2 molar ratios in the urea synthesis reactor and consequently a relatively higher conversion, without the drawback of the water build up in the carbamate recycle solution, mentioned before.

The combined effects of an increase in reactor "efficiency" and conversion have the obvious effect of decreasing the amount of the unconverted carbamate to be decomposed per pass, and thus of decreasing the utility consumption of the urea plant and of reducing the size of the equipment handling the carbamate recycle.

For instance an existing plant. originally designed and constructed on the basis of a 65 % conversion per pass, produced about 30 % more urea at 72 % conversion due to the fact that the specific steam consumption and heat load of a 72 % conversion urea synthesis loop is equivalent to about 67 % of the specific heat load of a 65 % conversion urea synthesis loop. Of course the increase in production will normally require additional liquid NH_3 pumping capacity and additional CO_2 gas compression capacity. According to recent developments such additional CO_2 gas compression capacity is not required.

Heat recovery - See process flow diagram Fig. n°1

The unconverted ammonium carbamate and excess ammonia contained in the reactor effluent are separated from the urea product solution by the well known method of heating the reactor effluent at reduced pressure, decomposing ammonium carbamate to NH_3 and CO_2 gas, vaporizing the excess NH_3 and separating the NH_3 - CO_2 gas mixture from the degassed urea product solution.

One of the most commonly used methods of NH_3 and CO_2 gas recovery and reutilisation consists of cooling the gas mixture, reacting NH_3 and CO_2 to form carbamate, dissolving carbamate and part of the excess ammonia in water and pumping the aqueous ammoniacal solution of ammonium carbamate thus obtained back into the reactor for recovery. It is common practice in conventional urea processes to carry such decomposer gas condensation in indirect heat exchange with cooling water and to irreversibly dissipate all the heat of reaction into the cooling water system.

On the contrary, in the M.B. urea process such decomposer gas condensation is carried out in indirect heat exchange with the reactor feed streams : the carbamate recycle solution and the liquid ammonia feed streams are preheated to about 112 °C prior to introduction into the urea synthesis reactor.

It is normal practice in the conventional partial or total recycle urea processes to preheat the liquid NH_3 reactor feed to 50 - 70 °C for the purpose of maintaining the reactor temperature at the operating temperature level of 180 - 190 °C. Invariably such reactors in conventional urea processes are operated adiabatically, that means that the exothermic heat of reaction of NH_3 and CO_2 to carbamate is utilized in the reactor to bring the reaction mixture to the operating temperature. No excess heat is removed from such adiabatic reactors.

On the contrary, in the H.R. process low level excess heat is recycled back to the reactor via the reactor feed streams. Normally the reactor in the H.R. process would operate adiabatically at 190 °C by maintaining the carbamate recycle solution reactor feed stream at about 85 °C and the liquid ammonia reactor feed stream at about 50 °C. Due to the fact that excess heat is transferred to both streams instead, and that they are preheated to about 112 °C, all the excess heat added to the carbamate solution above 85 °C and added to the liquid ammonia feed stream above 50 °C must be obviously removed from the reactor if one wishes to maintain its operating temperature at the same level of 190 °C.

This excess heat is removed from the exothermic reactor in the H.R. process by methods used to remove excess heat from the so-called "once through" exothermic reactors operating on gaseous CO_2 and liquid NH_3 reactor feed streams alone, with no liquid carbamate solution recycle.

By a simple improvement in the design of the heat removed system from the reactor, it is possible in the H.R. process to economically produce steam at a pressure level sufficiently high to utilize such steam in the carbamate decomposer.

By this method, about 55 % of the heat required to decompose carbamate in the 20 - 25 kg/cm^2 pressure level decomposer is supplied by the steam produced in the reactor and about 45 % less cooling water is required within the urea synthesis section due to the above mentioned fact that less heat of NH_3 and CO_2 reaction to carbamate is rejected to the cooling water system.

As it will be explained below, further heat economies are attained by condensing the first decomposer gas in indirect heat exchange with condensate to produce low pressure steam, which is used to degas the urea product solution in the second decomposer and in the atmospheric concentrator.

Furthermore, there is still a sufficient amount of heat of reaction available in the first decomposer gas after the production of steam and reactor feed streams preheating, to concentrate under vacuum the 75 wt% urea product solution by indirect heat exchange.

Decomposition, absorption and water balance

As it was mentioned before, the reactor effluent contains the amount of urea formed from the stoichiometric amount of fresh CO_2 gas make up fed to the reactor, the stoichiometric amount of water formed from the reaction of carbamate to urea, the amount of water and carbamate recycled into the reactor with the carbamate recycle solution and excess ammonia.

The major part of the reactor effluent is let down in pressure to about 24 kg/cm2 and steam heated to about 158 °C in a forced feed vertical decomposer of a novel design, operating at a relatively high velocity of the process liquid through the heat exchanger tubes, for the purpose of suppressing the formation of undesirable by product biuret. Carbamate is decomposed to NH_3 and CO_2 gas and excess ammonia is vaporized. The resulting gas mixture, however, contains 14-16 mol % of water vapor due to the fact that the gaseous phase is in equilibrium with the hot urea solution separated from the mixture. Such a decomposer gas with a relatively high water vapor content is not suitable for direct condensation to a liquid phase because the excess water would be detrimental to the reactor conversion.

For this reason in the H.R. process the first decomposer gas separator is provided with a tray section, in which the relatively hotter decomposer gas is counter-currently contacted with a relatively colder stream of urea solution. Such a stream of urea is obtained by withdrawing a minor part of the reactor effluent stream, cooling it to about 120 °C by adiabatic flashing at about 24 kg/cm2 pressure and by feeding such flash-cooled solution out the separator trays.

In the counter-current contact the decomposer gas is cooled to about 125 °C and more than 50 % of the water vapor contained in the decomposer gas is condensed to liquid and mixed with the downcoming urea solution. Such urea solution is heated up by the condensing water vapor, partially degassed and mixed with the main stream of urea product solution degassed in the decomposer.

The resulting liquid phase contains all the urea formed in the reactor, all the stoichiometric amount of water, the major part of the excess water recycled to the reactor in the carbamate recycle solution and some residual ammonium carbamate and excess ammonia.

For total degassing the urea product solution is further let down in pressure to about 3 kg/cm2 and steam heated at about 115 °C in the second decomposer to decompose the residual carbamate and to vaporize the residual excess ammonia.

The second carbamate decomposer system is of analogous mechanical and process design to the above described first decomposer system with respect to control of water vapor content in the decomposer gas.

The degassed urea product solution is finally let down to about 112 °C. All the residual excess water is evaporated from the urea solution, so that the stream leaving the system contains all the urea formed in the reactor and only the stoichiometric amount of process water formed in the reactor, and the small amount of water eventually added to the urea synthesis loop from an external source. For this reason such urea product solution usually has a concentration of about 75-76 wt %.

The gaseous phase from the atmospheric concentrator is water cooled and condensed to a liquid phase in the third condenser at about 38 °C, and the resulting solution pumped into the second condenser operating at about 3 kg/cm2. The gaseous phase from the second decomposer is water cooled and condensed to liquid in the second condenser at about 40 °C. The resulting solution is pumped into the first absorber, as it will be explained further below.

The gaseous phase from the first decomposer separator is first indirectly cooled with condensate to produce low pressure steam, which is used in the second and in the third decomposers.

The first decomposer gas partially condenses to liquid and the resulting mixture of gas and liquid is further cooled in indirect heat exchange with the liquid NH3 and the carbamate recycle solution reactor feed streams.

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The mixture of the residual first decomposer gas still containing about 15 % of the original CO2 gas, and the liquid phase formed by cooling is further water cooled in the carbamate condenser at 85 °C to bring to completion the reaction of CO2 gas with NH3 to form carbamate.

The stream of solution form the second condenser, mentionned above is distributed between the three heat exchangers, heating liquid NH3, the carbamate recycle solution and cooling water respectively, for proper temperature control.

The impure uncondensed excess ammonia issuing from the carbamate condenser is rectified in a conventional tray column in which it is counter currently washed by a part of the second condenser solution first and then by a stream of pure liquid NH3 reflux. The purified gaseous excess ammonia is liquefied in a water cooled condenser preheated as explained above and recycled to the reactor.

The liquid phase formed in the carbamate condenser described above contains all the water and the carbamate respectively evaporated and decomposed in the first, second and third decomposer, and some excess ammonia. This solution is preheated in indirect heat exchange with the first decomposer gas as described above and recycled to the reactor, thus closing the urea synthesis loop.

At process equilibrium the amount of carbamate contained in the carbamate recycle solution is equal to the amount of carbamate in the reactor effluent (with somme allowance for losses of carbamate via the 75-76 wt % urea product solution) and the amount of water present in the carbamate recycle solution is equal to the difference between the total amount of water present in the reactor effluent and the stoichiometric amount of process water formed in the reactor by dehydration of carbamate to urea, subsequently discharged from the urea synthesis loop via the 75-76 wt % urea solution product, as described before.

Practically speaking, it can be assumed that at process equilibrium 100 % of the fresh CO2 gas make up reactor feed stream is converted into the stoichiometric amount of urea and process water, and that the amount of carbamate contained in the carbamate recycle solution reactor feed stream passes unchanged through the urea synthesis reactor, is then decomposed to NH3 and CO2 gas, recovered and recycled back to the reactor.

For practical purposes it can be also assumed that the amount of water contained in the carbamate recycle solution is mixed with the process water formed in the reactor, separated from such process water by evaporation in the decomposers, recondensed in the condensers and recycled to the reactor.

For the reason exposed above, the unconverted ammonium carbamate and the water recycled to the reactor are the basic and important constituents of the urea synthesis loop. At process equilibrium their ratio is constant and depending upon the operating pressure and temperature conditions in the carbamate condenser their ratio assumes definite values.

Such operating values are mainly dictated by the solubility and salting-out temperature of ammonium carbamate. An uncontrolled return of water to the carbamate condenser causes a variation in crystallization point of the carbamate recycle solution. The resulting synthesis loop instability and potential sources of mechanical problems due to carbamate crystallization are obvious.

Viceversa, the advantages of maintaining the return of water to the condenser and thus the H_2O to carbamate ratio constant, are also obvious : the possibility of crystallizing the carbamate recycle solution is eliminated, and the urea synthesis loop is firmly maintained in constant equilibrium.

Various methods are presently used to detect the variations in H_2O to carbamate ratio in the carbamate recycle solution. The most commonly used method is the direct chemical analysis of the solution. The drawback of this method is the fact that such analysis is tedious and time consuming, and its results are available only several hours from the time of sampling : in the meantime the urea plant operator

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has no available information on the relative trend of the H_2O to carbamate ratio values.

Other methods of using continuous analysers operating on the basis of the measurement of one of the various physical properties of the carbamate recycle solution as for instance viscosity, density or sound conductivity and relating these properties to the concentration usually prove to be relatively inaccurate and subject to mechanical problems.

Mavrovic succeeded in developing a novel method of a direct continuous and very accurate determination of the crystallization point of the carbamate recycle solution by means of a simple and conventional apparatus. The scale of such measuring apparatus is directly calibrated in temperature degrees, correlated with the crystallization point of the carbamate recycle solution. The repeatability and the sensivity, for a variation of carbamate content in solution, expressed in terms of equivalent CO_2, from 32.0 wt % to 32.5 wt % of CO_2 is visibly registered on the instrument temperature scale, with a relative error or deviation of 0.5-1 °C.

The continuous crystallization point measuring device is installed on the carbamate recycle solution reactor feed line and is set to automatically regulate the amount of water vapor returned to the carbamate condenser.

This proved in practice to be the most valuable aid to the urea plant operator in maintaining the urea plant on stream and under safe operating conditions for extended periods of time. Emergency shut downs and consequent raw material and product losses are practically eliminated, as well as the usual mechanical problems with the high pressure carbamate recycle pumps.

4 - RESULTS

The results of incorporating all the above mentionel improvements and features in the design of the H.R. urea synthesis process are quite remarkable. A table is given below for illustration.

Technip - Mavrovic H.R. urea Process

Industrial results

Raw material and utility consumption per metric ton of prilled urea produced :

Ammonia,	T/T	0.58
CO2	T/T	0.75
Steam -11 absolute bars-	T/T	0.50
Electric power	kWh/MT	135 *
Cooling water (12 °C T)	m3/MT	42

Note :
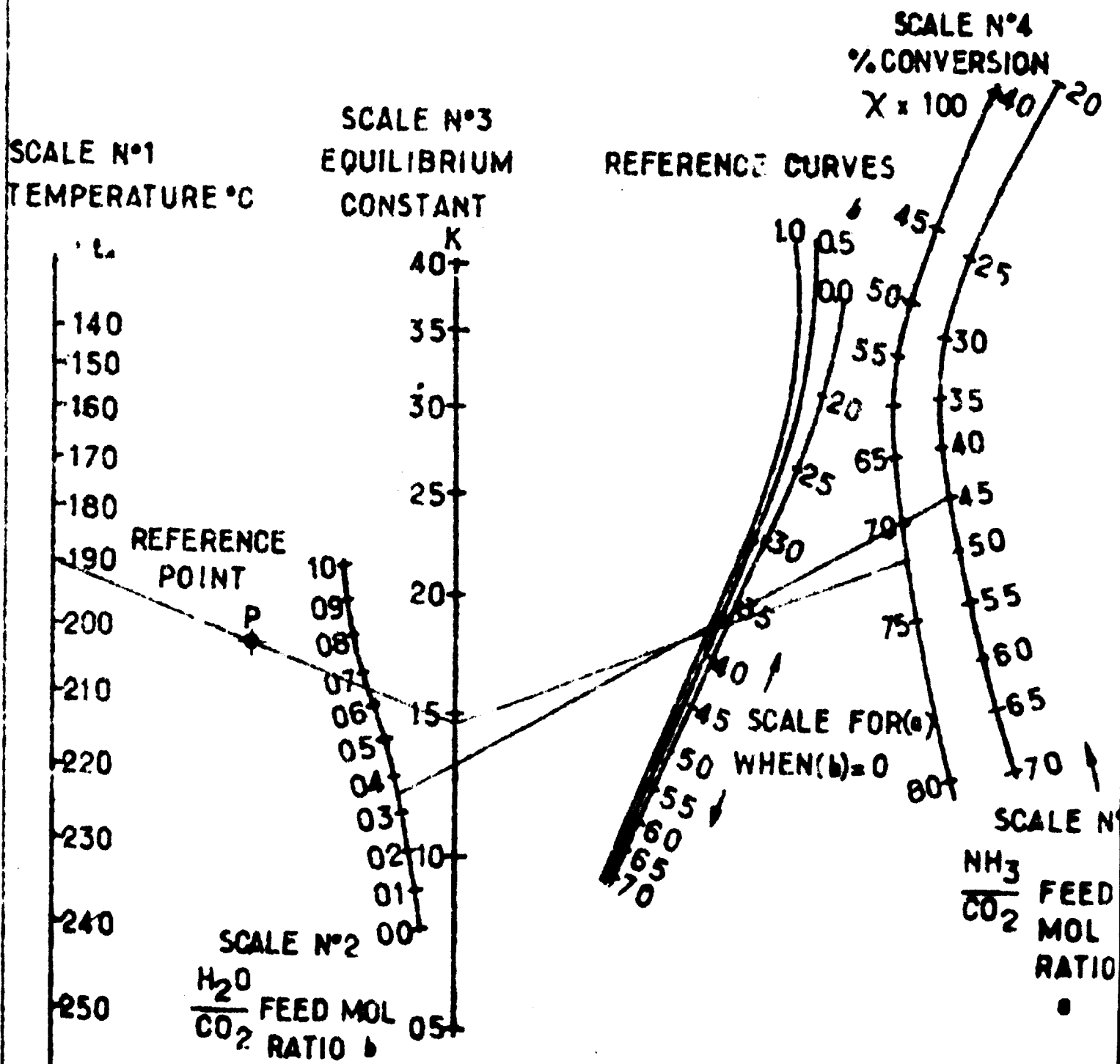
Acoording to recent process improvements and developments the electric power consumption is reduced to 90 kWh/Metric Ton.

References:

1. Patent U.S.P. 3,579,636   I. Mavrovic 1971 Heat Recycle Process

2. Patent U.S.P. 3,574,738   I. Mavrovic 1971 O_2 Introduction to Prevent Corrosion

EQUILIBRIUM UREA YIELD

SCALE N°4
% CONVERSION
X x 100

SCALE N°3
EQUILIBRIUM
CONSTANT
K

SCALE N°1
TEMPERATURE °C

REFERENCE CURVES

REFERENCE POINT
P

SCALE FOR (a)
WHEN (b) = 0

SCALE N°2
$\frac{H_2O}{CO_2}$ FEED MOL RATIO b
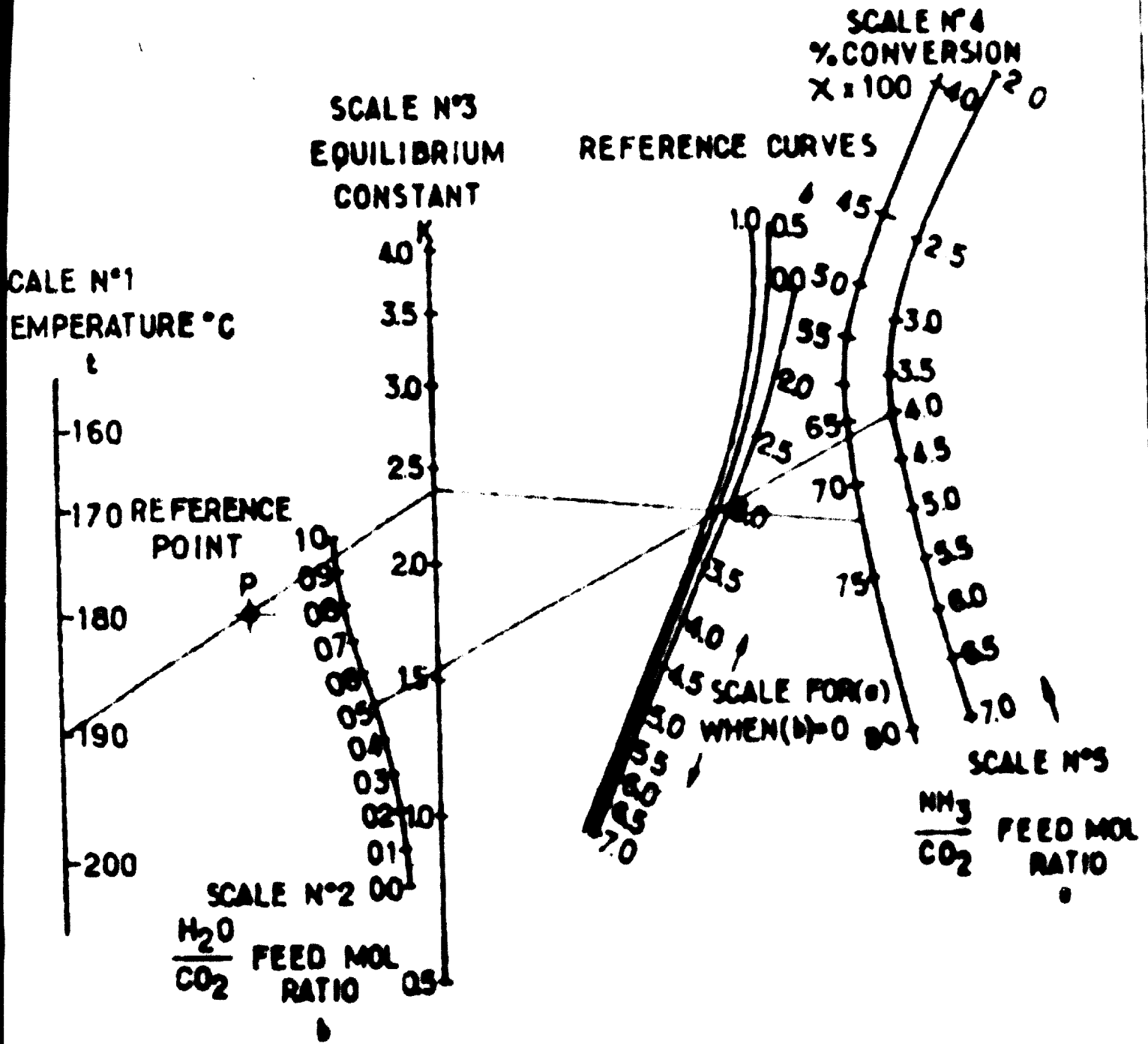
$\frac{NH_3}{CO_2}$ FEED MOL RATIO a

NOMO.GRAPH 1

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EQUILIBRIUM CONVERSION
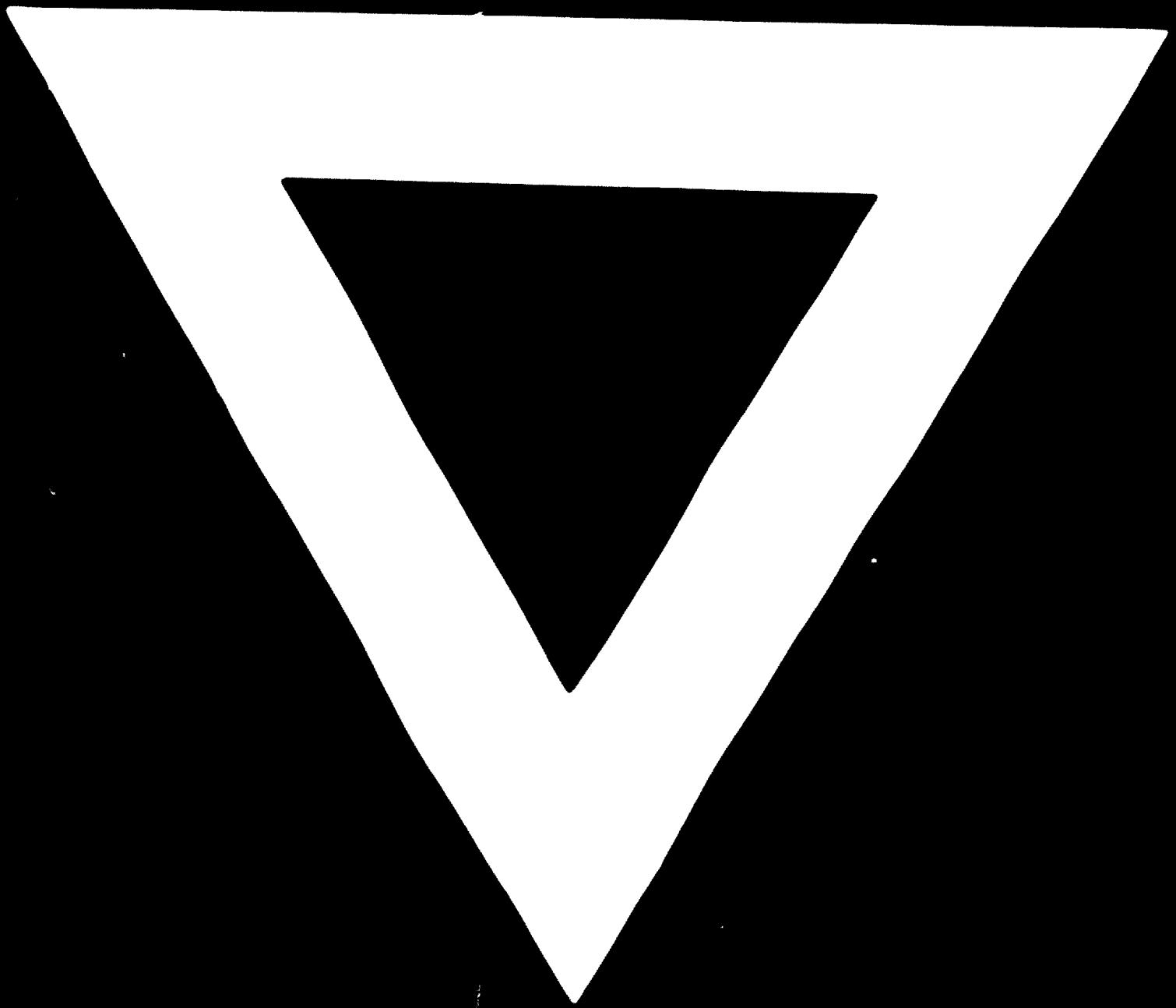OF CARBON DIOXIDE TO UREA

EQUILIBRIUM UREA YIELD

SCALE N°4
% CONVERSION
X x 100

REFERENCE CURVES

SCALE N°3
EQUILIBRIUM
CONSTANT

SCALE N°1
TEMPERATURE °C
t

REFERENCE POINT

P

SCALE N°2
$\frac{H_2O}{CO_2}$ FEED MOL RATIO
b

SCALE FOR(a)
WHEN(b)=0

$\frac{NH_3}{CO_2}$ FEED MOL RATIO
a

SCALE N°5

NOMOGRAPH 2

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EQUILIBRIUM CONVERSION
OF CARBON DIOXIDE TO UREA

8 . 8 . 73