where the integration constant I (1114.5) is determined from the known value of $H^{\circ} - H^{\circ}_{\circ}$ at 1183.16° K. The results are given in Table IV. Within the accuracy of the values listed, Equation 1 is satisfied exactly. The free energy change for the transformation of γ -iron to α -iron at a number of temperatures is given in Table V. Fisher (12) compiled a similar table by taking values (substantially the mean) from Johanson (14) and Zener (34).

Since nearly all reliable available data were used to establish the tables, very few independent checks could be made. Körber and Olson (21) have determined the enthalpy of liquid iron at 1600° C, relative to α -iron at 20° C, as 18,800 \pm 200 calories per gram atom. A slight extrapolation of Umino's data of Table III (subtracting 2.1 calories per gram to change the reference state from 0° to 20° C.) gives 18,500 calories; Table IV gives 18,350 calories. The enthalpy of iron at 1900° K. relative to its value at 298.16 given by Kelley (18) is 18,820 calories; Table IV gives 18,611 calories. The agreement with the data of Umino for γ -iron in the temperature range 910° to 1400° C. has previously been mentioned (see Table II).

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Catalytic Isomerization of Isomeric Hexanes

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The only commercial hexane isomerization units in this country were built by the Standard Oil Co. (Indiana) based on its process. The present work was undertaken to determine the manner and rate of isomerization of the hexane isomers to aid in establishing optimum conditions for these units.

The results indicate that isomerization of n-hexane to its isomers takes place in a stepwise fashion, but the steps do not take place with equal ease. The methylpentanes are first formed from *n*-hexane relatively slowly. The equilib-

NONSIDERABLE work has been reported on the isomeriza- \checkmark tion of alkanes (1). These investigations were concerned primarily with determining the composition of the equilibrium mixture of products that resulted under various reaction conditions. More recently fundamental work has been done on the mechanism of isomerization, particularly of butane (5-7, 9). Little has been published to reveal the path of an isomerization reaction when several products can be produced.

The five isomeric hexanes which coexist at equilibrium afford the simplest system for investigating the sequence in which the individual isomers are produced. Therefore, a systematic study of the individual hexane isomers under carefully controlled condi-

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rium between 2-methylpentane and 3-methylpentane is established rapidly. The rate of formation of 2,3-dimethylbutane from the methylpentanes is moderate. Neohexane (2,2-dimethylbutane) is difficult to form, but once formed it is extremely stable under isomerization conditions.

These data established that it was important to operate at close to the equilibrium octane number to obtain the maximum yield of neohexane in the commercial neohexane unit.

tions was undertaken wherein the conversion was varied over a wide range. By plotting conversion against the hexane product distribution and extrapolating back to zero per cent conversion according to the method of Schneider and Frolich (8), an insight into the initial reaction products should be obtained. From these data it should be possible to deduce the path of hexane isomerization. Portions of the present work have been cited (4) and a complete presentation of the results is provided here.

EXPERIMENTAL

The hexanes used in this work were purchased from the Phillips Petroleum Co. The n-hexane contained 96% n-hexane, 1% 3methylpentane, and 3% methylcyclopentane; the presence of



methylcyclopentane was not considered detrimental as it does not adversely affect the isomerization of paraffins (3). The 2-methylpentane after refractionation on a 100 theoretical plate column to remove a small amount of 2,3-dimethylbutane was $99\%^+$ pure. The 3-methylpentane contained 3% 2-methylpentane, and was used without further purification. The 2,3-dimethylbutane and 2,2-dimethylbutane were c.P. grade and used as received.

The aluminum chloride was a commercial grade obtained from the Hooker Electrochemical Co. and was used without further purification. The hydrogen chloride was commercial anhydrous gas produced by the Harshaw Chemical Co. Linde commercial hydrogen was used as received.

The experiments were made in a 1490-ml. reactor (2) the contents of which could be stirred under pressure at 1750 r.p.m. By circulating steam through a surrounding jacket, the reactor could be brought to reaction temperature in 5 minutes, and by circulating cold water it could be quenched in a corresponding length of time.

A standard run was made as follows: 700 milliliters of the selected hexane were placed in the reactor with 50 grams of aluminum chloride and 15 grams of hydrogen chloride. The reactor was pressured with hydrogen to 1000 pounds per square inch at the operating temperature of 100° C. in order to reduce side reactions to a minimum (3). The hexane and catalyst were stirred for the predetermined period of time at 100° C., then cooled and allowed to settle at 10° to 20° C. for sampling. The samples for analysis were withdrawn as liquid by means of a bleed-out tube which dipped below the surface of the liquid in the reactor.

The product was fractionated on a column packed with glass helices to remove any trace of butanes that may have been formed. Actually the amount of cracking to light hydrocarbons was negligible in all cases—1% or less. After being washed with ice water to remove any dissolved aluminum chloride, a 100-ml. sample was fractionated at a reflux ratio of about 100 to 1, on a 60 theoretical plate column packed with wire gauze. The accuracy obtained with this fractionation column is illustrated by the analysis of a known mixture, as shown in Table I. The analyses thus obtained were satisfactory when the original hexane was at least 20% converted to other isomers. In lower conversions it was necessary to rely mainly on analyses by the mass spectrometer. The products at conversion as low as 5% could be determined accurately by first concentrating the isomerized materials by fractionation and then analyzing the overhead cuts on the mass spectrometer.

The above standard procedure was followed in all except three cases. Two experiments on the methylpentanes were made at $^{1}/_{50}$ the standard amount of catalyst and one experiment was performed on 2,2-dimethylbutane at double the amount. The results with more and less catalyst were adjusted to the standard amount by proportion, thus making all results represented on the same plot comparable. Unpublished work from this laboratory indicated the rate of isomerization of paraffins to be almost directly proportional to the catalyst concentration.

REACTION PRODUCT OF ISOMERIC HEXANES

Sixteen experiments were made, the results of which are shown in Table II. When these data are plotted as actual conversion against the percentage of the various hexane isomers in the product (based on 100% converted), a set of curves is obtained which may be extrapolated to zero per cent conversion. This gives an insight into the initial reaction products which may be arbitrarily referred to as primary and secondary products depending on whether their curve intersects the ordinate at a positive value or passes through the origin.

n-Hexane. Figure 1 shows the curves obtained for n-hexane. These indicate that 2-methylpentane, 3-methylpentane, and 2,3-dimethyl-

butane are primary products and that 2,2-dimethylbutane is a secondary product. The composition of the primary products from the isomerization of *n*-hexane is: 51% 2-methyl-

TABLE I. FRACTIONAL ANALYSIS OF KNOWN MIXTURE						
	Composition, Vol. %					
	Found	Known				
2,2-Dimethylbutane 2,3-Dimethylbutane 2-Methylpentane 3-Methylpentane n-Hexane	$5.3 \\ 16.1 \\ 49.5 \\ 22.8 \\ 6.3$	5.616.148.424.25.6				



Figure 2. Conversion Rates of Isomeric Hexanes

		TABL	е II.	Isom	ERIZA	TION O	f Isom	eric H	IEXANE	S AT	100°	С.				
Hexane used			<i>n</i> -He	xane			2-Me pent	thyl- ane	3-1 p	Methyl entane	-	$Methyl-pentanes^a$	2,2-Dir bu	nethyl- tane	2,3-Din but	aethyl- ane
AlCl ₃ , wt. % of hexane HCl, wt. % of hexane			10 3	. 9 . 3			11.0 3.3	$egin{array}{c} 0.21 \ 1.1 \end{array}$	10.8 3.2	$10.8 \\ 3.2$	$\begin{smallmatrix}0.21\\1.1\end{smallmatrix}$	$\begin{smallmatrix}10.9\\3.3\end{smallmatrix}$	$\substack{11.0\\3.3}$	$22.0 \\ 6.6$	10.8 3.3	10.8 3.3
Contact time, min.	9	5	20	54	99	284	5	8	5	58	5	57	83	411	5	20
Composition of reaction product Butane, wt. % Debutanized liquid, vol. %	•••	• •		0.0	0.1	0.4	0.2		••*	1.3		1.1	0.0	0.5	0.1	0.5
Pentanes 2.2-Dimethylbutane 2.3-Dimethylbutane 2-Methylpentane 3-Methylpentane n-Hexane + residue	0.5 1.6 5.1 2.2 90.6	0.7 2.0 5.8 2.8 88.7	1.3 2.7 8.5 4.2 83.3	3.0 4.5 15.4 7.5 69.6	$2.9 \\ 6.1 \\ 17.0 \\ 8.3 \\ 65.7$	$\frac{8.4}{8.8}$ 28.6 13.2 41.0	$0.3 \\ 7.7 \\ 62.7 \\ 29.3 \\ 0.0$	0.0 2.2 79.4 18.3 0.0	2.5 11.5 52.8 31.4 1.8	$8.0 \\ 14.5 \\ 47.0 \\ 23.4 \\ 5.6$	0.0 0.6 9.6 89.8 0.0	$\begin{array}{c} 0.7 \\ 5.1 \\ 15.7 \\ 50.0 \\ 25.1 \\ 3.9 \end{array}$	$96.0 \\ 0.9 \\ 2.0 \\ 0.9 \\ 0.2$	${ \begin{smallmatrix} 0.8 \\ 62.6 \\ 5.7 \\ 18.0 \\ 8.3 \\ 4.6 \end{smallmatrix} }$	2.3 76.1 14.9 6.7 0.0	$\begin{array}{r} 0.2 \\ 4.4 \\ 46.9 \\ 31.6 \\ 16.6 \\ 0.3 \end{array}$
Conversion, liquid vol. $\%$	9.4	11.3	16.7	30.4	34.3	59.0	8.05	$2, 2^{b}$	15.85	28.15	0.60	24.7 ^b	4.0	36.6	23,9	53.1
Hexane product distribution, mole % 2.2-Dimethylbutane 2.3-Dimethylbutane 3-Methylpentane 8-Methylpentane n-Hexane ^a Mixture of 67% 2-methylpen ^b Conversion to products other	5.3 17.1 54.0 23.6 tane an than 2	6.0 17.8 51.0 25.2 ad 33%	7.7 16.3 50.5 25.5 3-metl	9.8 15.0 50.5 24.7 hylpenta	8.4 18.0 49.4 24.2 tane. .nes.	14.1 15.1 48.2 22.6	3.4 96.6 0	ióo 	15.6 73.0 11.4	28.1 51.9 20.0	0 100 0	20.6 63.5 15.9	23.5 48.9 22.4 5.2	15.7 48.9 22.8 12.6	9.5 62.1 28.4 0.0	8.4 58.8 32.2 0.6

pentane, 25% 3-methylpentane, and 18% 2,3-dimethylbutane. This is essentially the equilibrium proportion of these isomers found experimentally, calculated on a 2,2-dimethylbutane-free basis—namely, 55% 2-methylpentane, 25% 3-methylpentane, and 20% 2,3-dimethylbutane. The amounts of the primary products remain essentially unchanged to about 40% conversion, then begin to fall off slowly. The high conversion portions of the curves are drawn as broken lines because the experimental points are extrapolated to the equilibrium values (2), and the authors believed that there were not sufficient data in this region to fix adequately the sharp break, particularly in the 2,2-dimethylbutane curve.

It is not surprising that 2-methylpentane and 3-methylpentane are found as the primary products in about equilibrium concentrations, since these curves are extrapolated to zero from about 10% conversion. Figure 2 shows that 10% conversion of *n*hexane corresponds to a contact time of 10 minutes whereas only 2 or 3 minutes are required to establish the equilibrium between 2-methylpentane and 3-methylpentane. Analysis of the product from a *n*-hexane run, in which only 2 or 3% is converted, would be required to show other than the equilibrium ratio. The accurate analysis of such a small amount of isomerized product is extremely difficult since, in order to obtain an accuracy of 5% on the product curves in the region of 2 or 3% converted, it is necessary to know the composition of the isomer in the total product with an accuracy of $\pm 0.02\%$.

Although 2,3-dimethylbutane is indicated as a primary product, this is difficult to understand. The formation of the methylpentanes as primary products and of 2,2-dimethylbutane as a secondary product would lead one to believe that the isomerization of *n*-hexane proceeds in a stepwise fashion. Further studies at conversions less than 10% are needed to resolve this question.

2-Methylpentane and 3-Methylpentane. The methylpentanes are treated as a class because, as pointed out previously, 2methylpentane and 3-methylpentane isomerize to one another so readily that it is impossible to determine the primary products from either one individually. Figure 1 shows the primary isomerization product of the methylpentanes to be 100% 2,3dimethylbutane and 2,2-dimethylbutane as a secondary product. The 2,3-dimethylbutane curve decreases almost linearly with increase in conversion, whereas that for 2,2-dimethylbutane increases in the same fashion.

n-Hexane is indicated as a secondary product of the isomerization of methylpentane, although it would be expected as a minor primary product. The shape of the n-hexane curve is highly influenced by the one experimental point at low conversion, and this indicated the absence of n-hexane. It would require the presence of only a trace of *n*-hexane to indicate *n*-hexane as a minor primary product.

2,3-Dimethylbutane. As shown in Figure 1, the primary reaction products for 2,3-dimethylbutane are: 65% 2-methylpentane, 32% 3-methylpentane, and only 5% 2,2-dimethylbutane. *n*-Hexane is indicated as a secondary product. Although the amount is small, this is the only instance where 2,2-dimethylbutane is indicated as a primary isomerization product. The 2,2-dimethylbutane concentration increases as the conversion increases and reaches the equilibrium value of 42% at 100° C.

2,2-Dimethylbutane. Although 2,2-dimethylbutane is not readily formed, it is even less readily isomerized once it is formed. This makes it impossible to determine its primary isomerization products, because these products are isomerized to an equilibrium mixture of all the hexanes as quickly as they are formed. Nevertheless, the slope of the curve for 2,3-dimethylbutane in Figure 1 suggests that this isomer is the primary product.

ISOMERIZATION RATES OF ISOMERIC HEXANES

The various hexane isomers do not isomerize with equal ease. This is shown in Figure 2 where the per cent conversion of the various hexanes is plotted against the reaction time.

TABLE III. APPROXIMATE ISOMERIZATION HEXANES	RATES OF ISOMERIC
	Isomerization Velocity (k) , Hr. ⁻¹
$\begin{array}{l} n\text{-Hexane} \rightarrow 2\text{- and } 3\text{-methylpentanes} \\ 2\text{-Methylpentane} \rightarrow n\text{-hexane} \\ 2\text{-Methylpentane} \rightarrow 3\text{-methylpentane} \\ 3\text{-Methylpentane} \rightarrow 2\text{-methylpentane} \\ 2\text{-Methylpentane} \rightarrow 2\text{-methylpentane} \\ 2\text{-3Dimethylbutane} \rightarrow 2\text{-methylpentane} \\ 2\text{,3-Dimethylbutane} \rightarrow 2\text{,3-dimethylbutane} \\ 2\text{,2-Dimethylbutane} \rightarrow 2\text{,3-dimethylbutane} \end{array}$	$1.8 \\ 0.7 \\ 150 \\ 65 \\ 6.0 \\ 3.9 \\ 0.8 \\ 0.04$

The rates of conversion of 2-methylpentane and 3-methylpentane to their equilibrium mixture are both rapid. The equilibrium mixture at 100° C. contains 68% 2-methylpentane and 32% 3-methylpentane. The isomerization of 2,3-dimethylbutane is the next most rapid reaction, followed by that of *n*-hexane. Neohexane (2,2-dimethylbutane) is characterized by extreme stability, as it shows only about 3% conversion after a reaction time of 1 hour.

The isomerization rates for the various hexanes to the specific isomers have been calculated in terms of the specific reaction velocity, k, based on the assumption that the isomerizations follow a first-order reaction law using the equation:

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$$k = \frac{1}{t} \ln \frac{a - a_s}{(a - a_s) - a}$$

where a is composition of starting material, a, the composition at equilibrium, and a_t the quantity converted after time, t, in hours. Since there was considerable drift in the k values, the results are not exact but are of the right order of magnitude. These values are shown in Table III.

CONCLUSION

From the experimental data it is postulated that the hexanes isomerize in a stepwise manner:

> n-Hexane 2-Methylpentane $\stackrel{1|}{\underset{1}{\leftarrow}}$ 3-Methylpentane 2,3-Dimethylbutane 2.2-Dimethylbutane

The methylpentanes are rapidly interconverted; this prohibits deducing whether only one or both of the methylpentanes are intermediates in the transformation of *n*-hexane to 2,3-dimethylbutane. The rate-determining step in the formation of 2,2-dimethylbutane from *n*-hexane is the slow isomerization of 2,3-dimethylbutane to 2,2-dimethylbutane. In the isomerization of n-hexane to 2,3-dimethylbutane, the rate-determining step is the isomerization of *n*-hexane to the methylpentanes.

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Construction of Three-Dimensional Nomographs

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Use of three-dimensional nomographs for engineering formulas has not been tested in practical application, because nomographs of this type have been virtually nonexistent.

This article presents comparatively simple methods for construction of three-dimensional nomographs.

Although they present a convenient means of handling formulas with a proportionately larger number of variables than those handled by the more familiar two-dimensional nomographs, three-dimensional nomographs are more difficult to construct and to read. Nevertheless, in addition to the role they play as aids in computation, they provide a method of graphic representation which, as an aid to visualization, is difficult to achieve by three-dimensional graphs or two-dimensional nomographs.

THE construction of three-dimensional nomographs for certain types of formulas was described by d'Ocagne (5) as early as 1921. Virtually no application has been made of the methods which he presented; the reasons are apparently twofold: first, the theoretical discussion provided no simple means of adjusting the moduli of the several scales to accommodate variations in the ranges of the variables; and secondly, the method was applicable only to polynomials.

This article presents a simple theoretical approach to the construction of three-dimensional nomographs for all formulas capable of representation in a nomograph with a planar index surface. The hyperbolic coordinate system on which the construction is made may be modified to accommodate desirable variations in moduli and positions of the scales of the nomograph.

THREE-DIMENSIONAL HYPERBOLIC COORDINATES

XY-PLANE. The XY-plane is identical with the plane hyperbolic coordinate system described previously (2). Both principal axes are hyperbolic-i.e., they are of finite length-with points on the axes defined in terms of p's for which the corresponding distance of the point from the origin is p/(p+r); r is an arbitrary constant. The hyperbolic constants of the two principal axes, r_x and r_y , are independent.

Points in the plane are located with respect to points p_x and p_y on these axes by means of coordinate lines drawn to these points from the termini of the Y- and X-axes, respectively (Figure 1). Points with infinite abscissa and ordinate are located on an auxiliary \bar{Z} -axis passing through the termini of the X- and Yaxes, its origin on the X-axis and its terminus on the Y-axis. Points on this axis have the values of $p_{\bar{s}}$; the hyperbolic constant is r_i , where $r_i = r_y/r_x$.

VERTICAL AXES. The principal vertical axis is the Z-axis on the origin of the X- and Y-axes. Auxiliary vertical axes are the \overline{Y} -axis on the terminus of the X-axis, and the \overline{X} -axis on the terminus of the Y-axis (Figure 2). These axes are linear; their moduli have the relationships, $m_y^- = r_x m_z$ and $m_x^- = r_y m_z$. Points on these axes have the values q_z , q_y , and q_z , respectively.

VERTICAL PLANES. The vertical axes and the axes of the XY plane together determine three vertical planes; the XZplane, the YZ-plane, and the $\overline{Z}\overline{Y}$ -plane. Each of these planes is identical with the semihyperbolic planar coordinate system previously described (1), consisting of a single hyperbolic axis and two linear axes. Points in these planes are located in the manner shown in Figure 3: abscissas, by vertical coordinate lines; and