Organic and Biological Chemistry

Acidity of Hydrocarbons. XXI. Equilibrium Acidities of Some Hydrocarbons toward Lithium Cyclohexylamide in Cyclohexylamine[1]

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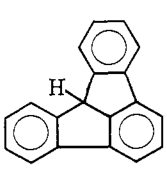
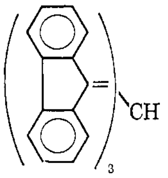
Abstract. Equilibrium acidity values are presented for ten hydrocarbons whose acidities are greater than the solvent, cyclohexylamine. Spectral measurements were made at room temperature of solutions of pairs of hydrocarbons competing for limited amounts of lithium cyclohexylamide. Relative to 9-phenylfluorene whose pK is taken as 18.49, the following pK values are derived: 3,4-benzfluorene, 19.38; 1,2-benzfluorene, 19.97; indene, 20.24; benzanthrene, 20.67; 4,5-methylenephenanthrene, 22.60; fluorene, 22.83; 2,3-benzfluorene, 23.16; 1,1,3,3-tetraphenylpropene, 26.2; 1,1,3-triphenylpropene, 26.4.

Until recently, few quantitative acidity values were available for hydrocarbons.[3] The literature abounds with qualitative and semiquantitative observations relating to syntheses, relative ease of metallations, etc., which, along with base-catalyzed proton exchange rates, have led to qualitative orders of equilibrium acidities and quantitative measures of kinetic acidities. For the derivation of the acidity constant of a hydrocarbon we require a knowledge of the relative amounts of the hydrocarbon and its conjugate base in a suitable equilibrium. Two general types of procedure are available.

In one method, the relative amounts of conjugate acid and base are determined in solvents of varying acidity or basicity. Aqueous solutions at various pH are particularly important but have not generally been used for hydrocarbons because of insufficient solubility. Even the extension to alcoholic or aqueous alcoholic media has been limited to such comparatively acidic hydrocarbons as fluoradene (I, pK = 13.5),[4] tris(biphenylenevinyl)methane (II, pK = 6.2),[5] and related derivatives of fluorene.



I



II

The extension of the pH scale to solvents of high acidity *via* Hammett's H_0 acidity function concept[6] has allowed the quantitative estimation of basicities of many weak bases.[7] The extension of the acidity function to basic media is expressed with the H_- scale and in recent years values for several solvent systems have become available.[8] Most of these solvent systems, however, have limited basicity and are suitable only for comparatively strong hydrocarbon acids. An example is the pK of 9-phenylfluorene (9-PF) determined by this method as 18.49 ± 0.1 in aqueous sulfolane,[9] 18.38 in aqueous dimethyl sulfoxide,[10] and 18.59 in ethanolic dimethyl sulfoxide.[10] Dimethyl sulfoxide media have been used for the determination of pK's of several more weakly acidic hydrocarbons.[11] One important advantage of the H_- approach is that with some reasonable assumptions concerning ratios of activity coefficients, the derived acidity values are related to a water system.

In the second general method, an acid in a suitable solvent is treated with the conjugate base of another acid and the relative amounts of the two acids and conjugate bases produced at equilibrium are measured. This method provides a direct measure of the *difference* in acidity of two compounds as expressed in the equilibrium

$$HA + B^- \rightleftharpoons HB + A^- \qquad (1)$$

$$K = \frac{[HB][A^-]}{[B^-][HA]} \qquad (2)$$

This method was utilized years ago by Conant, Wheland, and McEwen.[12,13] In their typical procedure, a hydrocarbon was mixed with an organosodium or -potassium in ether or benzene and the position of equilibrium 3 was estimated by color or by quenching

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[1] (a) This work was supported in part by the Directorate of Chemical Sciences, Air Force Office of Scientific Research, Grants No. 62-175 and 64-554; (b) a preliminary communication has appeared: A. Streitwieser, Jr., J. I. Brauman, J. H. Hammons, and A. H. Pudjaatmaka, *J. Am. Chem. Soc.*, **87**, 384 (1965); (c) paper XX: A. Streitwieser, Jr., R. A. Caldwell, R. G. Lawler, and G. R. Ziegler, *ibid.*, **87**, 5399 (1965).

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with carbon dioxide. On the basis of these early experiments with several hydrocarbons, McEwen gave estimates of pK values which are only now being made more quantitative.[1b,3,10,11]

$$HA + B^-M^+ \rightleftharpoons HB + A^-M^+ \qquad (3)$$

This paper reports our studies of several hydrocarbons using the second general method with lithium as the gegenion and cyclohexylamine as the solvent. All of the hydrocarbons studied were more acidic than cyclohexylamine so that equilibrium 4 lies far to the right.

$$RH + LiNHC_6H_{11} \rightleftharpoons R^-Li^+ + C_6H_{11}NH_2 \qquad (4)$$

Hence, a mixture of two such hydrocarbons in cyclohexylamine when treated with an insufficient amount of lithium cyclohexylamide (LiCHA) produces equilibrium 3, M = Li. The ultraviolet and visible spectrum of the mixture was compared with the spectra of the individual lithium salts in cyclohexylamine. From these spectral determinations and the known amounts of starting hydrocarbons the equilibrium constants could be determined.

Experimental Section

Materials Used. Some of the hydrocarbons used are or were available commercially and were purified by crystallization, chromatography, zone melting, or *via* the picrate, until they gave one peak on glpc. The ultraviolet spectra were generally identical with the literature, and the hydrocarbons had the following physical constants:[14] fluorene (F), mp[14] 116.5–117.5° (lit.[15] mp 116–117°); 11H-benz[*a*]fluorene (1,2-benzfluorene, 1,2-BF), mp 191.5–192° (lit.[16] mp 185.4–186°); 7H-benz[*c*]fluorene (3,4-benzfluorene, 3,4-BF), mp 123.2–123.6° (lit.[17] mp 124–125°). 4H-Benz[*def*]fluorene (4,5-methylenephenanthrene, 4,5-MP) was obtained in low purity from different commercial sources; the availability of this compound is variable. Purification *via* the picrate, mp 165–167° (lit.[18] mp 166°), gave product having mp 116.5–117.5° (lit.[19] mp 114.3–115.3°). This material showed one peak on glpc whereas the commercial product showed two peaks even after repeated recrystallization. Indene (In) was vacuum distilled under nitrogen and purified *via* the picrate. The product was further purified by preparative glpc followed by bulb-to-bulb distillation on the vacuum line and stored in small capillaries in a freezer. After storage for 1 month, glpc analysis still showed only a single peak. 11H-Benz[*b*]fluorene (2,3-benzfluorene, 2,3-BF) was prepared by the dehydrogenation of the tetrahydrobenzofluorene[20] with Pd–C,[21] mp 208.5–209.5° (lit.[20] mp 204–206°). Benzanthrene (7H-benz[*mn*]anthracene, BA) was prepared in low yield by reduction of benzanthrone with lithium aluminum hydride and aluminum chloride; alumina chromatography and recrystallization from ethanol gave mp[22] 83.5–84.5° (lit.[23] mp 80–82°). 9-Phenylfluorene was prepared from phenylmagnesium bromide and fluorenone followed by hydrogenolysis with Pd–C, mp 149–149.5° (lit.[24] mp 145°). 9-Phenylxanthene (9-PX) was prepared in a similar manner from xanthone and gave mp 147.8–148.1° (lit.[25] mp 145°).

1,3,3-Triphenylpropan-1-ol, prepared by reduction of β,β-diphenylpropiophenone with lithium aluminum hydride, was dehydrated by warming a solution of 10 mmoles in 2 ml of 20% sulfuric acid in acetic acid for several minutes.[26] The resulting 1,3,3-triphenylpropene (TriPP) was recrystallized, mp[22] 95–97° (lit.[27] 98–99°).

1,1,3,3-Tetraphenylpropene (TetraPP) was prepared following Garbisch,[26] mp[22] 126.5–128° (lit.[26] 126.5–127°).

10-Phenyl-9,9-dimethyldihydroanthracene (PDDA). An ether solution of 700 mg of 10,10-dimethylanthrone[28] was added to excess phenylmagnesium bromide. The product, 9-phenyl-10,10-dimethyldihydroanthran-9-ol, isolated in the usual way, was recrystallized from isooctane, mp[22] 167.5–168.5°. A solution of 677 mg of this alcohol in 25 ml of acetic acid was refluxed with 500 mg of 5% Pd–C and 2 g of zinc dust for 10 hr. After standing overnight, the mixture was filtered, diluted with water, and extracted with petroleum ether (bp 40–60°). The washed and dried organic extract was evaporated; recrystallization of the product from petroleum ether gave 405 mg, mp 147.5–148.5°.

Anal.[29] Calcd for $C_{22}H_{20}$: C, 92.9; H, 7.1. Found: C, 93.2; H, 7.2.

Spectra. Spectral data for most of the lithium salts of the hydrocarbons in cyclohexylamine were reported earlier.[1b,30] Data for the remaining hydrocarbons are as follows with λ_{max} mμ (ϵ) given: In, 371 (1263); tetraPP, 456 (29,800), 495 (18,400), 563 (36,800). The spectra were taken by mixing known amounts of hydrocarbon in cyclohexylamine with excess butyllithium in an inert atmosphere and were measured with a Perkin-Elmer 202 spectrometer. The carbanion spectrum generally developed immediately and remained stable for a sufficient period. In the case of tetraPP, however, the absorbance increased slowly, requiring about 0.5 hr to reach maximum absorbance.

Equilibrium Constants. The final procedure used was developed over the course of several years.[31] Brauman's runs differed most by making extensive use of drybox techniques, but most of the equilibrium measurements were made with minor modifications of the following procedure. The vessel used consisted of a cylindrical flask carrying two stopcocks, two Pyrex absorption cells (about 1-cm and 1-mm path length), and a stoppered vertical side arm. One stopcock carried a ground joint for attachment to the vacuum line and the second was fitted with a serum cap. For each run the apparatus was baked out. The solid hydrocarbons were added to the side arm. When solvents were used they were pumped off on the vacuum line. Cyclohexylamine was transferred in, and the entire system was pressurized with argon and mixed. Indene, being a volatile liquid, was introduced in a capillary flask together with a magnetic stirring bar to crush the vial at an appropriate time. Using a Hamilton gas-tight syringe, 1.5 *M* butyllithium in hexane was added through the serum cap, the solution was again mixed, and the spectrum was taken. The apparatus was scaled to fit within the cell compartment of the Perkin-Elmer 202 spectrometer. The intensity of the anion spectra generally diminished gradually with time and repeated scans were made. For each scan the absorbances were used to calculate the amounts of the two carbanions present. From the known amounts of hydrocarbons used, the equilibrium constant was calculated for each scan.

Table I summarizes the experimental results. This table records the *initial* quantities of the two hydrocarbons used and the average values of the organolithium concentrations found during the run. The error assigned to the equilibrium constant is the average deviation of K values calculated during the run. The number of spectral scans taken during the run is also given. The more acidic hydrocarbon is listed first. All measurements were at room temperature.

Results and Discussion

Validity of Results. The experimental results in Table I may be reduced to the average values in Table II. The errors assigned to the values are estimates based on the deviations from the mean results given during individual runs. The experimental errors in the extinction coefficients used to estimate carbanion concentra-

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(14) Corrected melting points taken on a Köfler micro hot stage.

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(31) We are indebted to Dr. M. Feldman for some preliminary experiments.

Table I. Equilibrium Comparisons of Hydrocarbons with LiCHA

Hydrocarbon, HA			Hydrocarbon, HB				
Symbol	Starting concn, 10^4M	LiA, 10^4M	Symbol	Starting concn, 10^4M	LiB, 10^4M	K^a	n^b
3,4-BF	37.8	2.56	1,2-BF	22.9	0.48	3.75 ± 0.3[c]	4
	37.8	28.9		22.9	10.25	4.25 ± 0.3[c]	4
	139.8	1.64		269.9	0.83	3.7 ±0.1[c]	3
	139.8	19.5		269.9	11.35	3.7 ± 0.1[c]	3
	172.6	0.6		425.7	0.37	3.7 ± 0.2[c]	3
	172.6	21.5		425.7	14.15	3.9 ± 0.2[c]	3
	32.8	3.58		37.6	1.05	4.28 ± 0.11[d]	5
	30.65	13.68		36.9	5.48	(5.0 ± 0.4)[e]	2
4,5-MP	40.2	15.2	2,3-BF	41.1	5.92	3.5 ±0.1[c]	3
	31.8	10.6		31.5	3.82	3.72 ± 0.3[d]	9
4,5-MP	15.6	1.32	F	68.4	3.62	1.55 ± 0.1[d]	2
	9.0	0.97		39.4	2.54	1.74 ± 0.03[d]	3
	14.9	1.03		77.2	2.85	1.92 ± 0.36[d]	15
2,3-BF	0.709	0.442	TriPP	151	0.123	2000 ± 800[d]	29
	0.549	0.310		149	0.113	1700 ± 600[d]	33
9-PF	153	1.42	1,2-BF	381	0.131	30.5 ± 4.9[d]	2
	107	1.86		923	0.668	29.6 ± 1.0[d]	7
3,4-BF	2.77	1.26	4,5-MP	101	0.0994	(870 ± 500)[d]	8
	1.45	0.86		514	0.366	(2200 ± 1000)[d]	18
	48.4	2.10		2558	0.950	1670 ± 90[e]	3
	7.71	2.15		1917	0.559	1320 ± 80[e]	6
	9.165	2.73		2358	0.787	1340 ± 90[e]	6
	6.36	2.22		1676	0.686	1290 ± 80[e]	6
2,3-BF	2.6	1.995	TetraPP	76.1	0.141	2100 ± 600[e]	5
	14.91	2.99		674	2.45	690 ± 150[e]	4
1,2-BF	16.61	1.17	In	199.5	7.84	1.86 ± 0.1[e]	5
In	105.5	8.04	BA	12.3	0.294	3.36 ± 0.1[e]	4
3,4-BF	32.89	2.29	BA	34.0	0.16	15.7 ± 0.4[e]	4
	17.23	2.00		28.25	0.238	15.5 ± 0.4[e]	11
In	81.5	9.23	4,5-MP	788	3.67	274[e]	1[f]

[a] $K = ([HB]_0 - [LiB])[LiA]/\{([HA]_0 - [LiA])[LiB]\}$. Values in parenthesis were not used in computing the averages in Table II. [b] Number of spectral scans. [c] By J. I. B. [d] By J. H. H. [e] By E. C. [f] Solution was stable and did not change with time.

tions probably contribute insignificant error compared to the effect of decomposition of the solutions. The pairs of compounds were so chosen that the spectral analysis of the carbanion mixtures was straightforward; that is, anions with well-separated absorption peaks were used.

Table II. Average Values for Equilibrium Constants

HA	HB	K	log K
3,4-BF	1,2-BF	3.9 ± 0.2	0.59 ± 0.02
4,5-MP	2,3-BF	3.6 ± 0.1	0.56 ± 0.02
4,5-MP	F	1.7 ± 0.1	0.23 ± 0.03
2,3-BF	TriPP	1800 ± 600	3.26 ± 0.15
9-PF	1,2-BF	30 ± 2	1.48 ± 0.03
3,4-BF	4,5-MP	1400 ± 100	3.15 ± 0.03
2,3-BF	TetraPP	1000 ± 300	3.00 ± 0.15
3,4-BF	BA	15.6 ± 0.3	1.19 ± 0.01
1,2-BF	In	1.86 ± 0.1	0.27 ± 0.02
In	BA	3.36 ± 0.1	0.53 ± 0.02
In	4,5-MP	274	2.44 ± 0.01

On adding butyllithium to the cyclohexylamine solution of the hydrocarbons, in general, reaction to produce the organolithium compounds was virtually instantaneous. Butyllithium reacts rapidly with cyclohexylamine to give LiCHA, and the reaction of LiCHA even with triphenylmethane, a less acidic hydrocarbon than those studied here, is rather rapid.[32] With tetraPP, however, the color of the carbanion developed comparatively slowly, and stable spectra with excess base sometimes required 0.5 hr. Yet this hydrocarbon is much more acidic than cyclohexylamine. A tenfold range of concentrations gave constant extinction coefficients. An abnormally slow rate of reaction of tetraPP with LiCHA may be rationalized by steric hindrance. Models show that the tertiary hydrogen in tetraPP is buried within a wall of phenyl groups and is sterically inaccessible. This explanation is subject to experimental test, and we expect to study the phenomenon in further detail.

The spectra of the equilibrium solutions generally showed a gradual decay with time, probably from reaction of the organolithium derivatives with the traces of oxygen and dissolved silicone grease present in the system. The carbanion concentrations were often of the order of 10^{-4} M, and mere traces of oxygen are sufficient to account for the decay of color. In some larger scale experiments, hydrocarbon recovered from treatment with butyllithium in cyclohexylamine apparently contained oxidized material. Although LiCHA reacts with oxygen, the amount present at these equilibria is so minute that the bulk of the reaction must be directly with the carbanion. Generally, these decomposition reactions were sufficiently slow that repeated scans could be taken and an equilibrium constant could be determined for each spectral scan. This decomposition removes material from the equilibrium, but the amounts involved were usually very small. In some cases, particularly between hydrocarbons having acidities differing by a few powers of ten, the more acidic hydrocarbon was present in very small

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amount and errors due to oxidation or decomposition are more significant. One criterion that such oxidation does not introduce a large error is the constancy of the K values calculated for each scan during a run. The average deviation of K was usually about 10% or less.

In some runs the variations are much greater. The slow reaction of tetraPP with LiCHA caused a problem in that formation of carbanion was concurrent with destruction. Equilibrium constants for this hydrocarbon and also for triPP have large uncertainties. Significant change occurred during the period of one spectral scan, and K values calculated at the beginning and end of a run varied by a factor of 2. Equilibrium studies of triPP with PDDA were especially bad. Different runs gave K values varying by several orders of magnitude, and even during a single run K would vary from less than unity to greater than unity. As a result, the acidity value reported previously[1b, 3] for PDDA is in error, although the derived qualitative conclusions are not much affected.

Different carbanions apparently react with oxygen with widely varying rates. The initial reaction is probably an electron transfer[33, 34]

$$R^- + O_2 \longrightarrow R\cdot + O_2\cdot^-$$

Similar equilibrium studies with cesium as the cation gave solutions which were qualitatively more stable than with lithium cation.[35]

For most of the hydrocarbon pairs studied, the principal evidence that the derived equilibrium constants do not have large systematic errors lies in the constancy of K values in experiments with different concentrations of hydrocarbons and carbanions and in the reproducibility of the several systems studied by more than one experimenter using somewhat different techniques. We conclude that the probable errors assigned on the basis of average deviations are realistic in order of magnitude.

Acidity Order. The hydrocarbon pairs studied overlap so that the order of acidities in Table III can be constructed. In a procedure of this sort errors can accumulate, but two sets of redundancies show that this accumulation is not serious. The direct measurement of BA relative to 3,4-BF gave log K = 1.19 ± 0.01; the sequence 3,4-BF, 1,2-BF, In, BA gave log K = 1.39 ± 0.04. The direct measurement of 4,5-MP relative to 3,4-BF gave log K = 3.15 ± 0.03; the sequence 3,4-BF, 1,2-BF, In, 4,5-MP gives log K = 3.30 ± 0.04. In both cases the differences are somewhat larger than the estimated combined errors and are in the same direction. In compiling the "pK" values in Table III, the average values were used. These values are assigned relative to the Langford and Burwell value of 18.49 for 9-PF.[9,10] The relative values are probably good to ±0.1 unit except for triPP and tetraPP, which may be in error by ±0.2 unit.

We have termed this table an "acidity order" and have referred to "carbanions" although it should be recognized that the conjugate bases involved are actually lithium carbanide ion pairs. Fluorenyllithium has been shown to exist primarily as ion pairs in cyclohexylamine.[36] Furthermore, a simple interpretation of the equilibrium order in Table III implies the assumption that the same type of ion pairs are involved for each of the alkyllithium compounds studied. Hogen-Esch and Smid[37] have presented evidence recently for the existence of two types of ion pairs with alkali metal fluorenyls. In ethers that are good cation solvators (*e.g.*, tetrahydrofuran), fluorenyllithium exists mostly as the solvent-separated ion pair at room temperature. The bathochromic shift in the visible spectrum of lithium compared to cesium fluorenyl in cyclohexylamine suggests that the former exists as the solvent-separated ion pair.[38] The possibility that both types of ion pair are present can be checked by a study of the effect of temperature on the spectrum and such studies are planned. Meanwhile, this ion-pair problem means that care must be taken in the use of the data in Table III. This problem appears to be less important for cesium compounds; fluorenylcesium exists entirely as contact ion pairs in all of the solvents studied[37] and is probably a contact ion pair in cyclohexylamine. The compounds in the present paper have also been studied with CsCHA and further discussion is deferred to the following paper.[35]

Table III. Acidity Order of Hydrocarbons toward LiCHA

Hydrocarbon	Symbol	Relative log K	pK
9-Phenylfluorene	9-PF	0	(18.49)[a]
3,4-Benzfluorene	3,4-BF	−0.89 ± 0.04	19.38
1,2-Benzfluorene	1,2-BF	−1.48 ± 0.03	19.97
Indene	In	−1.75 ± 0.04	20.24
Benzanthrene	BA	−2.28 ± 0.05, −2.08 ± 0.05	20.67
4,5-Methylene-phenanthrene	4,5-MP	−4.04 ± 0.06, −4.19 ± 0.05	22.60
Fluorene	F	−4.34 ± 0.07	22.83
2,3-Benzfluorene	2,3-BF	−4.67 ± 0.07	23.16
1,1,3,3-Tetraphenylpropene	TetraPP	−7.7 ± 0.2	26.2
1,1,3-Triphenylpropene	TriPP	−7.9 ± 0.2	26.4

[a] From ref 9 and 10.

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[38] This conclusion differs from the interpretation presented in ref 30.