LANTHANIDE SHIFT REAGENTS IN THE CHEMISTRY OF HETEROCYCLIC COMPOUNDS. A REVIEW

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After the pioneering work of Hinckley [1] and Sanders and Williams [2], the lanthanide shift (shifting) reagent (LSR) method in NMR spectroscopy was rapidly taken up by many chemists. By the middle of 1977, the number of studies carried out with the aid of LSR had reached the thousands, among them being some forty reviews [3-34, 450-457, 504-509] and a monograph [35]. In this list, approximately every fourth study deals with heterocycles. They and the data on which this review is based embrace the literature over the years 1969-1978.

The ability of paramagnetic ions to induce shifts in NMR signals was known even before Hinckley's discovery but had not been widely used. The chief merit of the lanthanide induced shifts (LIS) is that their magnitude is directly dependent upon the geometric structure of the molecules and solution. Another advantage of the lanthanide reagents is the insignificant broadening of the lines in the NMR spectra. This is particularly important when an LSR is used to simplify complex spectra and to determine hidden spin—spin interaction constants. Chiral LSR, interesting modifications of lanthanide shift reagents, are opening new possibilities for stereochemistry.

The theoretical and experimental bases of the LSR method will not be described in this review. They may be found in earlier reviews on the subject.

HETEROCYCLES INVESTIGATED BY THE LSR METHOD

In Table 1, studies in which the LSR method was used in the NMR spectroscopy of heterocycles are listed systematically.

The largest number of publications deal with nitrogen heterocycles. Pyridine, quinoline, and their derivatives were among the first nitrogen-containing substrates on which the effect of the new lanthanide reagents were tried [4, 46, 54, 116, 160-162, 350, 494], the mechanism of the effect of lanthanides on NMR spectra established [115, 117, 122, 123, 128, 134, 147, 148, 151, 153, 156, 157, 172, 173, 174, 462, 466], x-ray structure determinations [351-353, 467] and magnetic investigations of the adducts [354-356, 407] carried out, the connection between the magnitude of the induced shifts and the geometric structure of the adducts elucidated [109, 118, 124, 126, 130, 138, 144, 150, 154, 158, 159, 404, 468], and the stoichiometry determined [120, 121, 129, 135, 140, 405]. Isoquinoline [115, 117, 125, 128, 149, 150, 152, 159, 163, 203, 469],, acridine [115, 126, 133, 159, 191], naphthridines [115, 136, 144, 495], phenanthrolines [115, 124], phthalazines [115, 180, 181, 409] and other azines [104, 115, 178, 179, 182-185, 206, 410, 417], azafluorenes [142, 145, 168, 169], and piperidines [46, 86, 137, 189, 198, 216, 400, 411, 412, 471] have also been studied in detail. There are data on phenanthridine [115, 164], pteridine [115], morpholine [195, 214], quinuclidine [46, 118, 209, 413, 415, 472], and a number of other condensed and bridged structures. Among the five-membered nitrogen heterocycles which have been studied are pyrroles and pyrrolidines [46, 54, 56, 72, 86, 89, 109-111, 388, 400, 462, 463], pyrromethanes [47, 70, 71, 73], indoles [64, 65, 67, 69, 78, 86, 391, 392, 402, 460, 462], isoindoles and indolizines [69], cyclazine [66,459], imidazoles and benzimidazoles [49-59,75,81,88,89,388], pyrazoles and pyrazolines [52,56,74,76,87,103-105,388,462], oxazoles and ozazolines [60,81,98,389, 395], oxadiazoles [82], thiazoles and benzothiazoles [61, 62, 77, 81, 90, 465], tri- and tetrazoles [75, 80, 108, 388], porphyrins [48, 68, 390, 461], azapropellanes [84, 99], tropanes [393], and several other bridged and condensed systems. There is evidence of the

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TABLE 1. A Digest of Literature References to LSR Studies of Heterocycles[a]

Heteroatom	Heterocycle type	Coordination Center: heteroatom of the heterocycle	Coordination Center: carbonyl	Coordination Center: OH or COOH	Coordination Center: amino group	Coordination Center: N	Coordination Center: —O—	Coordination Center: S=O, SO_2, N=O, N—O or P=O	Coordination Center: anion	Lanthanide base of the LSR;[b] Pr	Lanthanide base of the LSR;[b] Gd	Lanthanide base of the LSR;[b] Dy	Lanthanide base of the LSR;[b] Yb	Lanthanide base of the LSR;[b] more than two	Chiral LSR
1	2	3	4	5	6	7	8	9	10	11	12	13	14	15	16
N	Aziridines	36, 381—383, 458; 37 (2)	38		36, 39			382							383
	Azetidines		40—44, 384—387	357	45			112		44				43	
	Azoles	46—48, 459; 49—59, 89, 388, 462 (2); 388 (3), (4); 60, 389 (O); 61, 62 (S)	47, 63—73, 390, 460; 74—76 (2); 75 (3), (4); 389 (O); 63, 77 (S)	461	78, 391, 462	392; 79 (2); 80 (3); 81 (2), (O), (S)		82 (2, O); 80 (3)		56, 57, 79, 390	462	54	46, 89	47, 48, 389	78
	Nonaromatic, five-membered heterocycles	46, 54, 83—86, 89, 99, 393, 394; 36, 87, 88 (2); 396 (2, O, P); 395 (O); 397 (O, P); 90 (S); 398 (P)	84—86, 91—97, 100, 101, 393, 399, 400; 76, 103—105, 464 (2); 42, 98, 401 (O); 42, 43 (S); 398 (P)	106, 107, 393, 394, 402	108 (3); 465 (S)	50, 109—111, 463		403 (O, S); 113 (O, P); 112 (S)	111, 463	98, 99, 395, 464		54	46, 89, 100, 402	43	90, 103, 113, 397, 401
	Azines with one heteroatom	4, 46, 54, 79, 80, 86, 89, 109—111, 114—159, 161—163, 165—169, 350—356, 404—407, 462, 466—469, 492—495	66, 69, 164, 170			57, 142, 167, 459		4, 80, 148, 155, 171—175, 469	111, 176, 177, 408, 470	57, 79, 155—159, 174, 177, 408, 470	161, 162, 350, 462	54, 492	46, 89, 163, 492	147—154, 172, 173, 404, 407, 466, 468	164
	Other azines	115, 178—181, 409 (2)	85, 182—185 (2)			85 (2)	182 (2)	410 (2)		181, 409					183, 184
	Nonaromatic, six-membered heterocycles	46, 85, 86, 118, 137, 160, 186—191, 391, 393, 411—415, 471—473; 83, 192, 194 (2); 103 (2, O); 195 (O)	41, 65, 67, 85, 86, 102, 118, 186, 187, 196—205, 393, 400, 416, 496; 100, 103, 104, 199, 200, 206, 417 (2); 401 (O); 43, 386, 387 (S)	107, 189, 207—209, 393, 402	204; 45 (S)		210—213; 214 (O)	215, 216, 418, 419; 420 (O, P)		160, 201, 411, 471	200		46, 100, 202, 402, 496	43	103, 199, 200, 213, 401, 411, 414, 420
	Heterocycles with seven or more atoms in the ring	83, 193, 218, 219 (2); 48 (4); 224, 225 (2O, Si)	85, 93, 96, 98, 202, 220—222, 416; 196, 197, 223, 421 (2); 68, 390 (4); 205 (O)	107, 207, 208			210	215, 217, 418		98, 219, 222, 390			202	48	

O	Epoxides	32, 226—241, 381, 382, 422—429, 471, 474—478	228, 232, 235, 429	242				382		32, 471, 475, 478			240, 241		238, 239, 471, 477
	Oxetanes	243, 244													
	Five-membered heterocycles	46, 84, 86, 99, 195, 229, 245—250, 290, 430—433, 478, 479; 251—259, 480 (2); 251, 260 (S); 398 (P)	72, 84, 94, 186, 195, 223, 228, 231—233, 256, 261—281, 434, 435, 481—484, 498—500; 98, 389, 401 (N); 86, 198, 256 (2)	242, 268, 282—288; 107, 284 (2)	436, 473	60, 81, 389, 395 (N); 397 (N, P); 396 (2, N, P), (2, P)	228, 231, 232, 246, 254, 256, 289, 437; 252 (2)	419, 436; 403 (N, S); 113 (N, P); 82 (2N); 486 (2)		98, 99, 195, 250, 290, 389, 431, 432, 478, 483	287		46, 280, 286	430	113, 397, 401
	Six-membered heterocycles;[c]	210, 246, 259, 291—299, 438, 501; 214 (N); 195 (N), (S); 103 (2N); 252, 290, 438 (2)	114, 195, 232, 241, 262, 265, 272, 278, 300—317, 434, 440—442, 487; 401 (N); 201, 256, 262 (2); 489 (S)	299, 311, 312, 318, 441, 488	443	114	232, 291, 309; 252, 290 (2)	420 (N, P); 321, 324—327, 444, 446 (2, P); 320—323 (2, S); 319, 445 (S)	470	195, 201, 290, 315, 316, 323, 438	298		241, 317	314	103, 297, 313, 401, 420, 489
	Heterocycles with seven or more atoms in the ring	259, 501; 330, 439 (3); 328 (3), (4), (5); 329 (6)	205 (N); 98, 262, 276; 330, 439 (S)	437	224, 225 (2, N, Si)		232, 437	331 (2, P)		98					
S	Number of atoms in heterocycle 3	381						381							
	4						214	332, 333							
	5	86, 333, 334	72, 77, 94, 195, 270, 273, 274, 330, 334—336, 439; 42, 63, 77 (N); 337, 447, 502 (2)	288; 338 (2)	45; 90, 465 (N)	165, 473; 61, 62, 81 (N)	251, 260 (O)	84, 86, 175, 245, 339—342, 419, 486, 490, 503; 112 (N); 403 (N, O); 343 (2)							90
	6 or more	333, 344; 219 (2)	195, 314, 330, 334, 439, 491; 330 (O), (2); 439, 489 (O); 43, 386, 387 (N)		448; 45 (N)	195	195	298, 339, 449, 503; 319, 445 (O); 320—323 (2O)		43, 195, 219, 323, 347				298, 314, 344	489
P	Number of atoms in heterocycle 4						345								
	5		346; 398 (N)			398 (N); 397 (N, O); 396 (2N, O); 398 (O); 396 (2O)		347—349; 113 (N, O)		347					113, 397
	6							348; 420 (N, O); 321, 324—327, 331, 444, 446 (2O)							420

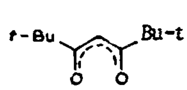
[a]Reference numbers separated by commas correspond to studies in which data are given for a single type of heterocycle with the same number of the same heteroatoms in a ring; the number of heteroatoms (if more than one) and/or their symbols (if more than one kind) are given in parentheses for the given ring. [b]Europium shift reagents are encountered in almost every study and so reference numbers for these studies are not listed separately. [c]This review does not include studies of carbohydrates.

interaction with LSR of β- [40-44, 112, 384-387], γ- [86, 91, 93-95], δ- [196, 197, 201-203], and ε-lactams [202, 220-222]. Several studies have been devoted to the aziridines [36-39, 381, 383, 458] and azetidines [40-45, 357]. Of LSR studies of five-membered heterocycles with oxygen atoms, the majority have been carried out on furans and benzofurans [46, 72, 94, 195, 232, 242, 254, 256, 261, 265, 266, 269, 270, 273-275, 277, 280, 282, 286-288, 290, 419, 473, 481-483, 485], di and tetrahydrofurans [46, 84, 86, 99, 195, 223, 229, 232, 245-250, 262, 263, 276, 278, 279, 281, 289, 430-432, 436, 437, 478, 479, 484, 499], 1,3-dioxolanes [86, 107, 198, 251-259, 284, 480, 486], and γ-lactones [228, 231, 233, 246, 264, 267, 268, 271, 279, 283-285, 434, 484, 498-500]. From the number of six-membered, oxygen-containing heterocycles studied, there are di- and tetrahydropyrans [210, 246, 259, 291-302, 313, 315, 318, 434, 438, 443, 487], pyrones [195, 300, 314], chromans and chromones [295, 301, 306, 312, 314], flavones and isoflavones [303, 305, 307, 308, 310, 311, 316, 317, 440-442], coumarins [114, 272, 304, 309], dioxanes [201, 252, 256, 262, 290, 438, 501], δ-lactones [232, 265, 278, 300, 488], and xanthone [241, 307, 440]; of small rings, there are epoxides [32, 226-242, 381, 422-429, 471, 474-478] and oxetanes [243, 244]; and of macrocycles, trioxocane [253] and crown ethers [328, 239, 501]. Many of the enumerated heterocycles enter into the composition of natural products or are fragments of complex bridged or polynuclear systems. Sulfur-containing rings appearing in this list are several times less common than the oxygen-containing ones. There are thiirane [381], thiophenes [72, 77, 94, 195, 270, 273, 274, 288, 330, 336, 439, 490], thiophanes [86, 334, 335, 419, 473], 1,2-dithiolanes [337, 338, 343, 447, 502], thiapyrones and thiapyrans [195, 314, 334, 491], oxathians [195, 319, 445, 489], cyclic sulfoxides [86, 245, 332, 339, 342, 381, 419, 449, 486, 503] and sulfones [86, 245, 340, 341], and also some condensed and bridged structures. There is evidence that the LSR method is applicable to phosphorus-containing heterocycles: phosphetans [345], phospholes, phospholines, and phospholidines [113, 346-349, 396-398, 403], dioxaphosphanes [326] and cyclic phosphates [321, 324, 325, 327, 331, 444, 446] and phosphamides [420]. In two studies, mention is made of the silicon-containing heterocycle silatrane [224, 225]. Scarcely ever studied by the LSR method are silanol [497], selenophene [274], selenapyrone [314], and tellurophene [274]. Cases are known of the interaction of LSR with simple and quaternary salts of pyridine [111, 176, 408, 463, 470] and pyril [470], as well cyan dyes [177].

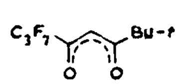
LSR CHELATES

At present, two types of lanthanide-based shift reagents have been established: LSR chelates and LSR salts. The former have a broad range of action and induce strong shifts in NMR signals, it is true, allowing spectra to be measured in a limited number of inert solvents. The latter have rarely been used so far and, as a rule, are used in studies of aqueous or aqueous acetone solutions. Consequently, they are often encountered in studies of peptides, carbohydrates, and other natural products which are insoluble in nonpolar solvents. They cause a much weaker shift in NMR signals than do LSR of the first type and will not be considered in this review.

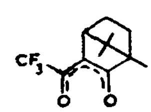
The overwhelming majority of spectroscopic problems of structural and conformational analysis of organic compounds can be, and has been, solved with the help of lanthanide chelates containing the radicals of these three β-diketones:



Dipivaloylmethane (DPM) or 2,2,6,6-tetramethyl-3,5-heptanedione (TMHD, THD)



1,1,1,2,2,3,3-Heptafluoro-7,7-dimethyl-4,6-octanedione (FOD)



3-(2,2,2-Trifluoro-1-oxoethylidene)-D-camphor (TFC)

LSR with other ligands are described in [48, 160, 358-361]. Chiral LSR contain optically active ligands [238, 239, 362-367, 369, 414]. LSR are encountered with partially deuterated ligands (for example, $Eu(FOD)_3$-d_{27}) [42, 96, 207, 249, 285, 306, 389, 421, 445]. For the synthesis of LSR, see [160, 238, 239, 358-360, 362-367, 370-374]. The list of solvents suitable for work with LSR chelates is extremely small and this restricts to a certain degree the range of their applicability. Most frequently used are CCl_4 and $CDCl_3$. Less commonly used are C_6D_6 [116, 411], CD_3CN [116, 175], and CD_3NO_2 [89]. Spectra have been taken at low temperature conveniently in CS_2 [127, 130, 135, 140, 406, 428]. The use of a nematic solvent has been described [132].

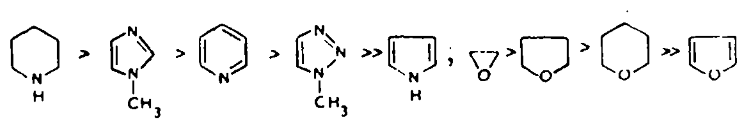
Among all the paramagnetic ions, apparently only europium allows one to observe the spin–spin splitting of NMR signals even at high concentrations of reagent. Among the other lanthanides which possess a good shift-broadening characteristic, praseodymium and ytterbium must be mentioned. About 13% of the total number of studies related to the use of LSR in investigating heterocycles involve these lanthanides.

LSR-SUBSTRATE ADDUCT

From the point of view of HSAB (Hard and Soft Acid Base) theory, LSR are hard acids and consequently will react more readily with hard bases. In other words, the ease of coordination of the lanthanides with heteroatoms should decrease in the series N, O > S, P > Se; this has experimental support [86, 219, 324, 335, 346]. Primary amines and alcohols react the most vigorously; nitro compounds, esters of trifluoroacetic acid, mercaptans, sulfides and disulfides, thioketals, phosphines and their thiooxides, silanes, halogen derivatives, nitriles, and hydrocarbons* are comparatively inert towards LSR. In the majority of cases, the activity of the functional groups decreases in a sequence such as:

$$NH_2 > OH > SO,\ PO > CO,\ NO > NHCO > -O- > COO > \geq N > -S- > CN$$

Some of the heterocycles can be arranged in a similar series of decreasing activity in reaction with LSR:



These series are derived by comparing the size of the LIS of structurally similar groups (for example, $-CH_2X$) or, better yet, by studying bifunctional derivatives and mixtures of monofunctional compounds [144, 195, 336]. The sequences given can be perturbed in cases where intramolecular hydrogen bonds are formed [211] or by the effect of steric impediments (LSR-chelates are acids with the coordination center surrounded by bulky ligands). For example, tertiary amines in the aliphatic and aliphatic-aromatic series react with LSR much more poorly than primary and secondary amines do and also more poorly than pyridine-type bases [109, 110, 158, 395]. This property of LSR is used in investigating polyfunctional compounds, for distinguishing the structures of isomeric molecules if they have different environments about the nucleophilic center [50, 51, 104, 105, 141, 142, 146, 167, 194]. The lack of an appreciable reaction of one of the isomers with the LSR is of itself sufficient for assigning the isomeric structures.

If the functional group contains two or more heteroatoms, complex formation proceeds through the most nucleophilic of them. Thus, amides and esters coordinate through the carbonyl oxygen atom [220, 221, 233, 270, 275]. Nitrosamines and nitroso compounds also coordinate through the oxygen atom [216, 217, 418]. Two spatially close coordination centers in a substrate molecule not uncommonly lead to the formation of bidentate adducts [75, 80, 85, 124, 208, 235, 256, 279, 299, 315, 340] even if the activities of the centers taken separately are quite different. If in solution there are several substrates (or an equilibrium mixture of conformers) or a basic molecule containing several functional groups, concurrent complex formation will occur and the observed NMR spectra will reflect the rates of reaction of the separate substrates or functional groups with the LSR.

The replacement of hydrogen, ^1H, by deuterium displays small (several percent) differences in the sizes of the induced shifts. Thus, deuteration of the methyl groups in methylpyridines allows one to detect this "isotope effect" [131, 143, 406].

*In order to bind olefins to LSR, their π-complexes with silver salts are used [34, 375].

SOME FEATURES OF THE APPLICATION OF LSR IN THE STUDY OF HETEROCYCLES

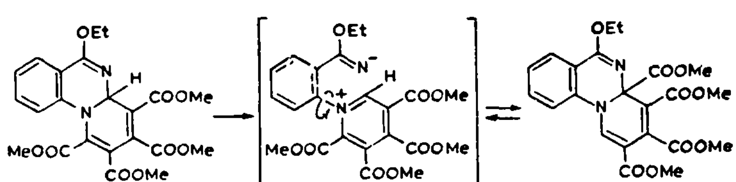
The highly selective nature of the action of LSR allows one to simplify complex NMR spectra. In many cases, such strong separation of the signals is obtained that the spectrum can be interpreted in the first-order approximation. The expansion of the spectrum can be easily controlled by the addition of various amounts of the LSR to the solution of substrate. The size of the LIS for protons bound to carbon can reach 25-30 ppm. If the problem is to obtain hitherto inaccessible spin-spin constants or to carry out a double-resonance experiment, then the origin of the induced shifts does not, in general, play a large role. What is important is the possibility of simplifying the spectra without an appreciable loss of resolution. However, if conclusions are to be drawn on the basis of the size of the induced shifts, then the nature of the LIS becomes of decisive importance. Methods of separating out the pseudocontact contributions to the induced shifts, necessary for quantitative calculations of structures of organic molecules in solution, are described in reviews [28, 31, 32, 455]. These also analyze the sources of error in calculating the Eu–N or Eu–O bond lengths in adducts on the basis of the size of the LIS (see also [62]). Methods of compensating for experimental errors in the treatment of the size of LIS can be recommended [376-379].

The simultaneous application of LSR and lanthanide broadening reagents (LBR, for a review see [380]) appears to be extremely useful [161, 162, 200, 287, 298], as do the use of the electronic-nuclear Overhauser effect and other methods for establishing the geometry of molecules in solution.

CHEMICAL REACTIONS INDUCED BY LSR

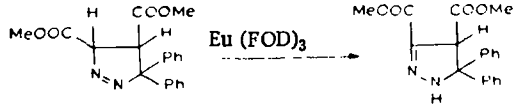
A number of cases have been noted in which the addition of an LSR to a solution of substrate having a nonrigid molecule leads to a change in the $^3J_{HH}$ or $^3J_{HP}$ constants [201, 208, 315, 325, 331]. This is evidence of the effect of complex formation on the conformational state or tautomeric equilibrium of the substrate molecules [396]. Other studies, on the other hand, have established the constancy of the constant J in the formation of adducts with LSR [84, 237, 240, 257], or the constancy of the conformational state [197].

There are two examples of the rearrangement of heterocyclic compounds in which LSR play the role of acid catalysts. Thus, the addition of $Eu(DPM)_3$ to a solution of compound I leads to the rearrangement to compound III via intermediate II. The existence of the II $\rightleftharpoons$ III equilibrium is confirmed by the build-up of one of the antipodes on the addition of $Eu(TFK)_3$ to racemate III which is clear from the gradual change in the PMR spectrum and the optical activity of the solution [199, 200].



I II III

The other rearrangement induced by LSR is the conversion of a 1-pyrazoline, IV, into the 2-pyrazoline, V [105].



IV V

Both reactions are also catalyzed by protonic acids.

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