IUPAC Recommendations

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Comprehensive definition of oxidation state (IUPAC Recommendations 2016)

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Abstract: Oxidation state (OS) is defined using ionic approximation of bonds. Two principal algorithms are outlined for OS determination in a chemical compound described by a Lewis formula or bond graph. Typical origins of ambiguous OS values are pointed out, and the relationship between OS and the d^n electron configuration of transition metals is commented on.

Keywords: bond graph; bond order; electronegativity; ionicity; Lewis formula; oxidation number; oxidation state.

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1 Preamble

Oxidation state (OS) gives the degree of oxidation of an atom in terms of counting electrons. It scales trends in redox and acid-base properties, as well as in physical properties such as magnetism, and is a key component when tracking the course of chemical reactions. Thus, the concept of OS is important, and so is an agreedupon definition of what OS is and the algorithmic manner in which it is to be calculated. In the absence of an actual definition, algorithms have thus far been used to define OS.2

This Recommendation proposes a definition of OS based on ionic approximation of chemical bonds, illustrated on a molecular-orbital (MO) scheme and deduced from electronegativity considerations (Allen's scale). Two algorithms are formulated for the determination of OS in molecules, ions, and extended solids, and are illustrated with examples. Limits, beyond which OS ceases to be well-defined or becomes ambiguous, are exemplified; moving outside these requires additional measurements, round offs, estimates, or plain postulates. Specific uses that justify a Nominal OS are also explained.

The present authors have published a comprehensive Technical Report on OS [1]. It provides a summary of previous work and discussions concerning OS, and gives numerous examples of the application and use of algorithms to determine OS.

2 Definition

A recommendation has a normative side that suggests what should be done and a practical side that reflects common use. The normative side is generic: OS is the atom's charge after ionic approximation of its bonds.

The atom's charge is the ionic charge; a valence-electron count relative to the free atom (OS is a quantitative concept that operates on counted electrons). Several criteria were considered³ for the sign of the ionic approximation [1], but only one of them is reflective of current use and common values of OS; the atom's contribution to the bonding MO, which is associated with the atomic-orbital energy as illustrated in Fig. 1.

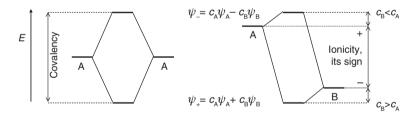


Fig. 1: The essence of ionic approximation based on contribution to the bonding MO. The mixing coefficients c_a and c_b refer to the atomic-orbital wavefunctions $\psi_{\mathtt{A}}$ and $\psi_{\mathtt{B}}$ in an LCAO-MO approach.

Figure 1 implies that, while A-A bonds are divided equally, the ionic approximation of A-B assigns the bond's electrons to the atom that contributes more to the bonding MO.4 Therefore, this contribution does not concern the electrons' origin upon bond formation (p. 1048, Ref. [1]), only their final allegiance. As Fig. 1 illustrates a concept, it is not to be taken as an instruction to start using quantum-chemical calculations to obtain OS.

¹ An alternative term «oxidation number» is also used in English. It is largely synonymous with OS, and may be preferred when the value represents a mere parameter or number, rather than being related to chemical systematics or a state of the atom in a compound.

² A set of postulated OS values for relevant elements remains a useful approach at the teaching level prior to bonding considera-

³ For example, polarity via dipole moment fails for molecules such as CO.

⁴ In general, electrons of each MO are assigned to their main atomic contributor.

On the practical side: In an AA bond, the two atoms are not always equivalent as Fig. 1 may imply. For example, if the NN bond in N₂O were extrapolated ionic, the charge⁵ of the central nitrogen would be +5 and the terminal one -3, suggesting unusual OS values. The definition is therefore narrowed to reflect the normal or common use of the term OS. Thus:

The oxidation state of an atom is the charge of this atom after ionic approximation of its heteronuclear bonds.

Bonds between atoms of the same element are not replaced by ionic ones; they are always divided equally.

3 Ionic approximation from electronegativity

In common use, the sign of the ionic approximation is obtained from electronegativities [2] rather than MO schemes. Of several scales discussed in Appendix B of Ref. [1], only the Allen electronegativity (Section 6.1) is truly independent of OS, since it relates to the average valence-electron energy of the *free* atom [3–5], as if the bonds implied in Fig. 1 were abstracted away (Fig. 2). The ionic approximation then assigns the bond electrons to the more electronegative partner.7

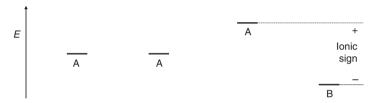


Fig. 2: Ionic-approximation sign according to relative energies of the free-atom valence orbitals, conveniently derived from Allen electronegativities.

The ionic approximation by electronegativity has a caveat due to the electron allegiance introduced in Section 2: When the more electronegative atom is bonded as a Lewis acid (a so called Z-type ligand, Ref. [6]), its acceptor orbital is at high energy. Under ionic approximation, the less-electronegative Lewis-base donor, typically a late transition metal, is therefore assigned the electrons it supplied to form the adduct bond. As pointed out in Ref. [7], such an electron allegiance can reveal itself *via* the adduct formation being reversible.

4 Algorithms

There are two closely related general algorithms for OS calculation:

4.1 Algorithm of assigning bonds

This algorithm works on Lewis formulas (p. 1026, Ref. [1]) of molecules and ions which show all valence electrons (Fig. 3). Its practical formulation uses electronegativity to deduce the ionic sign:

⁵ As a physical variable, the ionic charge of an atom (also known as the "charge number") is a number with sign preceding its value in units of elementary charge. As a nomenclature symbol in chemical formulas and names, ionic charge comes with trailing sign. Similarly, OS as a nomenclature symbol is in roman numerals.

⁶ MO schemes soon become complicated, making the ionic-sign allocation by orbital contributions or energies difficult and by atomic gross-population charges somewhat method dependent.

⁷ The ionic approximation by the A-B electronegativities is a simplification. When they are close (such as for H-P, H-Te, C-I or C-Se, Section 6.1), the ionic signs might be guided by analogies (see Section 5.5).

Oxidation state equals the charge of an atom after its homonuclear bonds have been divided equally and heteronuclear bonds assigned to the bond partners according to Allen electronegativity, except when the electronegative atom is bonded reversibly as a Lewis-acid ligand, in which case it does not obtain that bond's electrons.

Fig. 3: OS (in red) in CH_2F_2 (left) and in peroxynitrous acid (right), by assigning bonds to more electronegative partners on Lewis formula with all valence-electron pairs drawn as dashes.

The bond order of the homonuclear bonds will not affect OS, provided the isolated segment of all -AA-pair bonds, with their ionic-approximation signs, is symmetrical. Thus, while the OO bond order in Fig. 3 (right) is irrelevant, because the -OO- segment has a mirror symmetry, the NN bond order in an octet-fulfilling N₂O Lewis formula controls the OS: apply Fig. 3 to $\overline{\mathbb{N}}=\mathbb{N}=\overline{\mathbb{Q}}$ and $|\mathbb{N}=\mathbb{N}-\overline{\mathbb{Q}}|$ (p. 1038, Ref. [1]).

The caveat of the reversibly-bonded Lewis-acidic electronegative ligand (Section 3) is illustrated with the Rh–S bond in O_2S –RhCl(CO){ $P(C_6H_5)_3$ } $_2$ [8] in Fig. 4. The electronegative S atom, here a Lewis acid, does not keep the bond pair, the allegiance of which follows from the reversibility of this adduct back into SO_2 and RhCl(CO){ $P(C_6H_5)_3$ } $_3$.

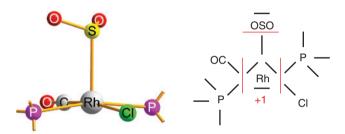


Fig. 4: OS of Rh by assigning bonds according to electronegativity, but invoking the caveat of reversibly bonded SO_2 as a Lewis acid, which therefore does not keep the bond electron pair: Rh does, with OS = +1.

4.2 Algorithm of summing bond orders

This algorithm works on Lewis formulas and bond graphs. A bond graph represents the infinite periodic network of an extended solid [9, 10]. It is constructed on a stoichiometric formula of the network's repetitive unit, with atom symbols connected by a line for each instance of the atom's bonding connectivity. Each line carries its own specific bond order.

Heteronuclear-bond orders are summed at the atom; as positive if that atom is the electropositive partner in a particular bond, and negative if it is not: the atom's formal charge (if any) is added to that sum, yielding the oxidation state.

In bond graphs of extended 3D structures, there are no formal charges.⁸ The obtained "ionized bond order sum", iBOS,⁹ equals OS directly as illustrated in Fig. 5 using AuORb, perovskite [11], where OS and bond

⁸ Formal charge is defined as the charge of an atom in a Lewis formula after $\frac{1}{2}$ of each of that atom's bonds has been assigned to it as electrons.

⁹ Named by analogy to the bond-valence sum, BVS, evaluated from structure data of extended solids.

orders follow from the 8-N rule at O, 8+N rule at Rb, and 12-N rule at Au. For a glossary of these rules, see p. 1027 in Ref. [1].

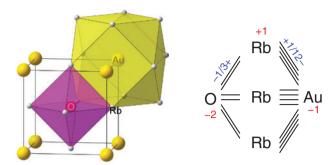


Fig. 5: Unit cell and coordination polyhedra of AuORb, perovskite, with its bond graph of ideal bond orders (values in blue) and OS (in red).

Lewis formulas may or may not have formal charges, as shown in Fig. 6. On the left, a formula with no formal charges yields OS = iBOS directly. On the right, the formal charge (the numerical variable FC) must be added to the sum at each atom; OS = iBOS + FC.

Fig. 6: OS (in red) by summing ionized bond orders (in blue) in Lewis formulas. Left: in CH₃F, directly, no formal charges. Right: in CO, with formal charges (in black, with trailing sign).

Bond orders in extended solids are not always obvious and may have to be estimated from bond lengths. This is done by converting each bond length into the so-called bond valence, which is a value entirely equivalent to the bond order in terms of two-electron bonds in molecules. The origins of the bond-valence approach, one ionic [12] and one covalent [13], are associated with Linus Pauling. In Ref. [13], an expression is given that evolved into the currently used relationship between bond valence and bond length:

$$BV_{ii} = \exp\left[\left(R^{0}_{ii} - d_{ii}\right)/B\right] \tag{1}$$

where BV_{ii} and d_{ij} represent the bond valence and the bond distance, respectively, between the atoms i and j. R^0_{ij} is the reference single-bond length parameter of the ij pair and B is a variable parameter often fixed at 0.37 Å (37 pm). This relationship makes it possible to determine OS directly from the bond distances obtained from crystal-structure data. For details, examples and relevant references, consult Ref. [1].

5 Epilogue

The applications of OS in chemistry are wide and deal with a cornucopia of chemical compounds and materials. It is therefore not surprising that, for some compounds, one value does not fit all uses, or that dedicated measurements or computations are needed to ascertain the actual OS. In those rare cases when the most convenient OS becomes a matter of choice, this fact must be clearly stated. Here we list the most important ambiguous situations. For more details and examples, see Ref. [1].

5.1 Non-innocent ligands

Redox-active ligands, called "non-innocent" after Jørgensen [14, 15], render OS less obvious when combined with a redox-prone central atom. Examples include complexes of O., NO, and dithiolenes, discussed in Ref. [1]. Drawing their Lewis formula requires information about bond orders from diffraction and spectral data. The OS of the transition-metal center can often be ascertained from spectral and magnetic measurements.

5.2 Metallic compounds

When bonding and antibonding orbitals/bands overlap in a metal, we are no longer entitled to make the ionic extrapolation implied by Fig. 1. There are simple metallic compounds of obvious OS, such as the golden TiO (+2), blue-black RuO, (+4), or coppery ReO, (+6), but, eventually, the assignment of conducting electrons to one of the bonded atoms has its limits. An example is AuNCa, perovskite [16], where neglecting its metallic character suggests Au³⁻ anions for which there is no support in theory.¹⁰ Such an OS is merely a practical value for redox balancing, like setting OS=0 for all metals in intermetallic phases of metallic character (not the semiconducting Zintl phases), or for those inner atoms in metal clusters that are solely bonded to other metal atoms.

5.3 Nominal OS

In systematic descriptive chemistry, OS is used to sort out compounds of an element; in electrochemistry, it represents the electrochemically relevant compound or ion in Latimer and Frost diagrams of standard (reduction) potentials. Such purpose-oriented OS may differ from the OS per definition and are termed nominal, or more specifically "systematic" or "electrochemical". Consider thiosulfate, the S₂O₃²⁻ anion: The sulfur–sulfur bond is practically a single bond, which yields OS = +5 for the central S and -1 for the terminal S atom by both algorithms (p. 1039, Ref. [1]). The electrochemical OS of sulfur in thiosulfate is their average, +2 (p. 1060, Ref. [1]). On the other hand, a systematic OS of +6 may be chosen for the central S atom, as if the sulfur–sulfur bond were approximated ionic, to emphasize the similarity of the peripheral O and S atoms obtaining OS = -2as in sulfate.

5.4 Nominal OS and redox reactions

As discussed by Vitz [17], redox reactions can be decomposed into two half reactions in which OS changes. Since $\Delta G^{\circ} = -nFE^{\circ}$, the standard reduction potential E° is available whenever we set up an OS to change in a half reaction. Consider the synthesis of $S_2O_3^{2-}$ from SO_3^{2-} and sulfur. We may see this Lewis acid-base reaction as a non-redox process by assigning a nominal OS = 0 to the thiosulphate terminal sulfur atom. Yet the same reaction can also be described as a redox one in terms of E° and the electrochemical OS = +2 of sulfur in thiosulfate. As a rule, if an oxidation takes place with an added oxidant, the OS should increase in the species that is being oxidized; if there is a reduction (with a reducing agent), it should decrease the OS in the species that is being reduced.

5.5 Need for choices, estimates, and round offs

Usage-related choices define the nominal OS, vide supra. Options arise also when the AB ionicity in Fig. 1 approaches zero and the distinction between positive and negative OS wanes. In Ref. [1] (p. 1054) this is

¹⁰ Ref. 16 suggests a 6s²-stabilized Au⁻ anion and 2e⁻ as in an electride.

illustrated with H₂PO₃; in textbooks the P-H bond electron pair is assigned to either H or P, yielding two OS alternatives. A similar example is CSe, where OS of -2 for Se is obtained via MO considerations or by analogy with CO₂.11 The same OS also suits ····C-Se-C··· compounds. For intermetallics of metallic character, the ultimate choice of OS = 0 is best if needed for redox chemistry.

Subtler estimates or round offs are required for compounds with electrons delocalized over nonequivalent atoms, manifested by a set of weighted resonance formulas; as an example in Ref. [1] for 1H-pentaazole on p. 1054, N_s^+ on p. 1038, or thiosulfate on p. 1039. Round offs are needed also in compounds that enter bonding compromises, as exemplified with S₄N₄ in Ref. [18].

Rounding is also necessary for bond-valence sums after the bond-length to bond-valence conversions with eq. 1. Their decimal values stem from the statistical distribution of bonding compromises in the set of compounds used to obtain the single-bond length parameter R_{ij}^0 in eq. 1.

On the other hand, reasonable fractions of small integers are obtained for OS in compounds such as B₆H₁₀ or B₁₀C,H₁₇ (p. 1040, Ref. [1]), or when two vicinal OS are indistinguishably mixed, such as for Fe in YBaFe₂O₅ (p. 1058, Ref. [1]), or when the ionic charge is distributed over several equivalent atoms, such as in $B_6H_6^{2-}$ (p. 1039, Ref. [1]) or I_3^- and N_3^- (p. 1037, Ref. [1]). OS in non-stoichiometric solids with randomly distributed defects is decimal: $NiO_{1.031}$ with interstitial oxygens has Ni of OS = +2.062, as does $Ni_{0.970}O$ with nickel vacancies.

6 Addendum

6.1 Allen electronegativity scale

Allen electronegativities in Pauling units [3-5].

Н									He
2.300									4.16
Li	Be		В	С	N	O		F	Ne
0.912	1.576		2.051	2.544	3.066	3.610		4.193	4.787
Na	0		Al 1.613	Si 1.916	P 2.253	S 2.589		Cl 2.869	Ar 3 . 242
0.869									
K	Ca		Ga	Ge	As	Se		Br	Kr
0.734	1.034		1.756	1.994	2.211	2,424		2.685	2.966
Rb	Sr		In	Sn	Sb	Te		I	Xe
0.706	0.963		1.656	1.824	1.984	2.158		2.359	2.582
Cs	Ba		Tl	Pb	Bi	Po		At	Rn
0.659	0.881		1.789	1.854	2.01	2.19		2.39	2.60
Sc	Ti	V	Cr	Mn	Fe	Co	Ni	Cu	Zn
1.19	1.38	1.53	1 . 65	1.75	1.80	1.84	1.88	1.85	1.59
Y	Zr	Nb	Mo	Tc	Ru	Rh	Pd	Ag	Cd
1.12	1.32	1.41	1.47	1 . 51	1 . 54	1.56	1.58	1.87	1.52
Lua	Hf	Ta	W	Re	Os	Ir	Pt	Au	Hg
1.09	1.16	1.34	1.47	1.60	1.65	1.68	1.72	1.92	1.76

^aVariation across the lanthanoid series has not been evaluated.

¹¹ This is in the opposite direction to the small electronegativity difference of 0.12, see Section 6.1.

6.2 OS and dⁿ configuration

The configuration d^n is a central-atom descriptor in transition-metal complexes. For a transition metal of Nvalence electrons, d^n yields OS as:

$$OS=N-n$$
 (2)

A problem arises when that central atom acts as a Lewis base and donates one of its d^n non- or anti-bonding electron pairs to an acceptor ligand (Lewis acid, a Z-type ligand [6]): Whereas OS remains the same (since the ionic approximation assigns the donated electron pair back to the Lewis base), the coordination geometry and magnetism at the central atom may no longer refer to the original d^n before that donation.

One of the examples discussed in Ref. [1] is the Au-B bond in Fig. 7 [19], where two Au d-electrons populate the weakly bonding MO so that Mössbauer spectroscopy still sees this MO's two electrons together with the rest of the d electrons on Au as d^{10} , suggesting OS = +1 for gold, in accord with the OS definition in Section 2. Yet the coordination at Au is square planar, typical of d^8 Au³⁺. The square-planar Au appears because the donated Au d-electron pair became the Au-B bond itself, and the geometry is now controlled by the energy minimum for the remaining 8 Au d electrons in the weakly antibonding MOs. To avoid the emerging ambiguity of the d^{10} "spectroscopic" versus the d^{8} "ligand-field" or "magnetic" configurations, a notation used by Parkin [6] may describe the central atom: Au of n=10 in d^{n-2} , where "2" symbolizes the weakly bonding "donated" d-electron pair, n enters eq. 2 for OS determination, and n-2 are the electrons in d orbitals subject to ligand field.

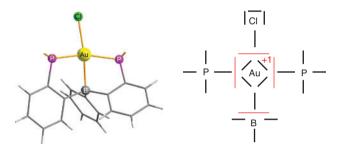


Fig. 7: Square-planar Au bonded to more electronegative B acceptor in a Z-type ligand [19].

In the same way for similar adducts of Z-type ligands and transition-metal Lewis bases: Rhodium in Fig. 4 has configuration d^{n-2} of n=8 that yields OS=+1 from eq. 2 (Rh is assigned the adduct-bond electron pair it donated) while the n-2=6 electrons at the low-spin 4d atom comply with the square-pyramidal coordination. More examples are found in Ref. [1].

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