# NO Bond length/stretching frequency relationships in C-nitroso compounds and their coordination complexes

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## Abstract

The mode of coordination of monomeric C-nitroso compounds to metals is discussed. In contrast to previous studies it is proposed that an understanding of the $\nu(N-O)/r(N-O)$ and $r(C-N)/r(N-O)$ relationships in the non-coordinated nitrosocompounds is of primary importance for assessment of the coordination mode. It is concluded that both $\nu(N-O)/r(N-O)$ and $r(C-N)/r(N-O)$ have linear interrelationships in C-nitroso compounds and that the coordination compounds of RNO have the same $\nu(N-O)/r(N-O)$ relationship as the non-coordinated monomers. Previous correlations of $\Delta\nu(N-O)$ with coordination mode are therefore correlations of $r(N-O)$ with coordination mode. $\sigma$-N and $\sigma$-O complexes of aromatic RNO conform to the same $r(C-N)/r(N-O)$ equation (within a very small error) as the non-coordinated monomers. The extent of deviation from the $r(C-N)/r(N-O)$ relationship for complexes of aliphatic RNO is of a similar order of magnitude to that which occurs when C-nitroso monomers form the *trans*-dimer. The coordination mode of aliphatic RNO is, with one exception, $\sigma$-N. Nitrosobenzene has a variety of coordination modes to transition metals but does not display $\sigma$-O coordination. *p*-Nitrosodimethylaniline undergoes $\sigma$-O coordination to $d^{10}$ metals.

## Introduction

Over thirty years ago, Lüttke [1] produced evidence for an inverse curvilinear relationship between NO bond length and the NO stretching frequency for a bond order variation ranging from one to three. Since that time there have been more measurements and in particular the advent of three-dimensional X-ray studies has increased the accuracy of bond length measurements and made it possible to extend our understanding of this interrelationship. To some extent this has been attempted by Talberg [2] who has also shown that in monomeric C-nitroso compounds the CN and NO bond lengths show a compensatory interrelationship [3,4]. It is necessary to consider (1) whether these relationships are still valid in the light of the most recent X-ray data, (2) whether gas phase bond length data can be incorporated into the

relationships established for solid state measurements and (3) to consider whether such interrelationships are modified by coordination of the *C*-nitroso compounds to inorganic and organometallic molecules or fragments. To the best of our knowledge this third question has never been explicitly addressed. It should be noted that *C*-nitroso compounds have a very wide range of coordination modes [5] illustrated in I–VII below and that a general correlation of coordination mode with the NO stretching frequency has been frequently employed as an aid to establishing which of the above modes is operative [6]. The use of such relationships is based upon the combination of X-ray crystal structure determinations with infrared spectroscopic measurements and three simple correlations have been widely used (a) an increase in $\nu$(N–O) on coordination is associated with $\sigma$-O bonding VII, (b) a small decrease in $\nu$(N–O) is associated $\sigma$-N bonding I, and (c) a large decrease in $\nu$(N–O) is associated with $\pi$(N–O) bonding II. We shall examine whether such generalisations are to be understood as a sub-set of the relationships first proposed by Lüttke [1] and Talberg [2] and consider the consequences for bonding in the complexes.
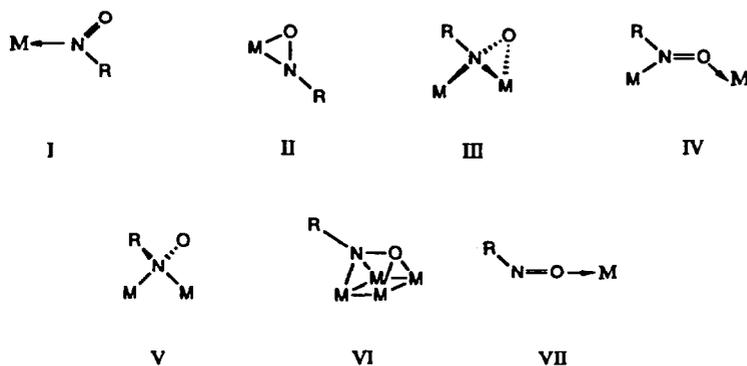


## Data and discussion

The relevant X-ray data and NO stretching frequencies for *C*-nitroso compounds are collected in Table 1, and in Fig. 1 we present the $\nu$(N–O)/$r$(N–O) relationship and in Fig. 2 the $r$(C–N)/$r$(N–O) relationship. By confining the molecules studied to monomeric *C*-nitroso compounds we are making use of a somewhat narrower range of NO-containing compounds than Talberg [7]. On the other hand we have been able to include many more *C*-nitroso compounds than were available to Talberg. From Fig. 1 and restricting the data to the cases where $r$(N–O) $\geqslant$ 1.19 Å we obtain a best straight line which is unaltered by the inclusion of gaseous monomers:

$$r(\text{N–O})/\text{Å} = 1.735 - 3.412 \times 10^{-4}\nu(\text{N–O})/\text{cm}^{-1}$$

This relationship is close to that obtained by Talberg whose relationship is also included in Fig. 1. It is apparent that geminally substituted nitrosocycloalkanes show some deviation from the above line. In Fig. 2 the $r$(C–N)/$r$(N–O) interrelationship is examined. Talberg has obtained the relationship:

$$r(\text{N–O})/\text{Å} = 1.150 + 0.016/\left[r(\text{C–N})/\text{Å} - 1.233\right]$$

Table 1

Structural data for *C*-nitroso compounds (all data relate to solid state apart from **S**, **T**, **V** and **W** which are gaseous)

| Compound | $r$(C–N)/ Å | $r$(N–O)/ Å | Ref. | $\nu$(N–O)/ $cm^{-1}$ | Ref. |
|---|---|---|---|---|---|
| 5-nitrososalicylic acid (**A**) | 1.426 | 1.234 | 7 | 1480 | 7 |
| 4-nitrosoanisole (**B**) | 1.467 | 1.228 | 8 | 1504 | 8 |
| ethyl-*m*-nitroso-*trans*-cinnamate (**C**) | 1.451 | 1.229 | 9 | 1480 | 9 |
| 4-nitroso-*N*,*N*-diethylaniline (**D**) | 1.38 | 1.27 | 2 | 1380 | 2 |
| | 1.42 | 1.25 | | | |
| 1,5-di-dimethylamino-4-nitrosobenzene (**E**) | 1.372 | 1.276 | 10 | 1332 | 11 |
| 10-bromo-2-chloro-2-nitrosocamphane (**F**) | 1.48 | 1.19 | 12 | | |
| 4-iodo-nitrosobenzene (**G**) | 1.43 | 1.21 | 13 | 1488 | 14 |
| 5-nitroso-4-phenethylbenzo[*b*]thiophene (**H**) | 1.419 | 1.213 | 15 | | |
| 2-methyl-6-nitroso-7-phenethyl-1,3-benzothiazole (**I**) | 1.41 | 1.23 | 16 | | |
| *trans*-1,4-dichloro-1,4-di-nitrosocyclohexane (**J**) | 1.505 | 1.139 | 13 | 1570 | 14 |
| 4-nitroso-5-triphenyl-phosphoranylidene-aminobenzo[1,2-C:3,4-*c*$^{-1}$]difurazan (**K**) | 1.369 | 1.265 | 17 | | |
| 9-nitrosojulolidine (**L**) | 1.452 | 1.227 | 18 | | |
| 1-chloro-1-nitrosocyclododecane (**M**) | 1.65 | 1.15 | 19 | 1578 | 20 |
| 9-cyano-2-chloro-2-nitroso-*trans*-decalin (**N**) | 1.542 | 1.121 | 21 | | |
| caryophyllene nitrosite (**O**) | 1.65 | 1.10 | 22 | 1578 | 22 |
| humulene nitrosite (conformer I) (**P**) | 1.509 | 1.133 | 23 | 1570 | 23 |
| (conformer II) | 1.530 | 1.191 | 24 | 1570 | 24 |
| 3,5-epidithio-2-nitroso-1,5-diphenylpenta-2,4-dien-1-one (**Q**) | 1.315 | 1.312 | 25 | | |
| *N*,*N*-dimethyl-3-iodo-4-nitrosoaniline (**R**) | 1.38 | 1.24 | 26, 27 | | |
| | 1.39 | 1.23 | | | |
| trifluoronitrosomethane (**S**) | 1.51 | 1.198 | 28 | 1595 | 29 |
| nitrosomethane (**T**) | 1.480 | 1.211 | 30a | 1564 | 14 |
| nitrosylcyanide (**U**) | 1.418 | 1.228 | 31 | 1501 | 32 |
| 4-nitroso-*N*,*N*-dimethylaniline hydrochloride (**V**) | 1.316 | 1.374 | 33 | 1049 | 34 |
| 2-methyl-2-nitrosopropane (**W**) | 1.517 | 1.202 | 30b | 1546 | 14 |

which clearly implies that $r$(N–O) cannot fall below 1.150 Å for a *C*-nitroso compound. Again the geminally substituted nitrosocycloalkanes cannot be incorporated within such an equation. The best relationship which includes all our collected data excepting **J**, **M** and **O** is

$$r(\text{N–O})/\text{Å} = 2.556 - 0.9269\ r(\text{C–N})/\text{Å}$$

The main purpose of these arguments is to make use of the $\nu$(N–O)/$r$(N–O) and $r$(C–N)/$r$(N–O) relationships in the cases of (a) dimerisation of *C*-nitroso compounds and (b) coordination by *C*-nitroso compounds. It is expected that in the former case the detailed change in electronic structure which occurs compared with that operative in the monomer is such that the $r$(C–N)/$r$(N–O) interrelationship will be totally altered. In Table 2 and Fig. 3 we make use of the bond lengths tabulated recently by Gowenlock and McCullough [35] together with a further value
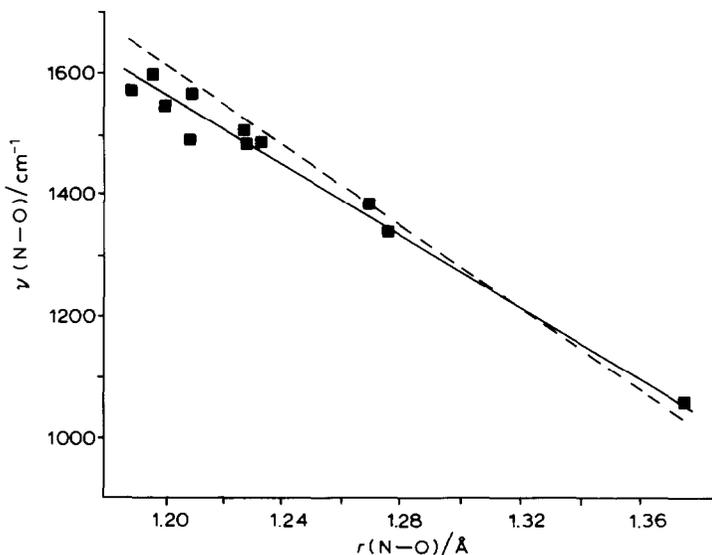
Fig. 1. Plot of N–O stretching frequency (wavenumber) against N–O bond length of C-nitroso monomers from data summarised in Table 1. ——— our line - - - - - - Talberg's line

[41] all of which are of a high degree of accuracy. It is seen that the use of the pair of electrons from the N atom of the monomer in the formation of the *trans*-dimeric $N_2O_2$ group fundamentally alters the $r(C-N)/r(N-O)$ interrelationship operative for monomers. This then directs attention to the question of coordination by C-nitroso monomers. If coordination to a metal, by whatever mode of those listed
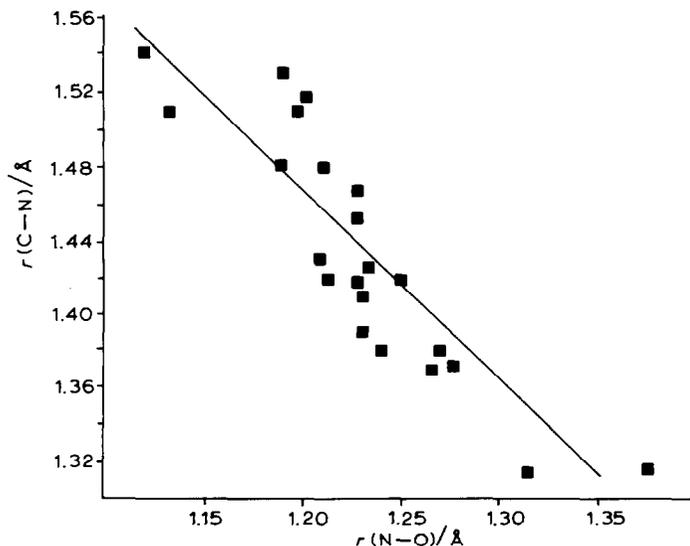


Fig. 2. Plot of C–N bond length against N–O bond length of C-nitroso monomers from the X-ray data summarised in Table 1.

Table 2

Structural data for *trans*-dimeric nitroso compounds, $(RNO)_2$

| R | $r(C-N)/\text{Å}$ | $r(N-O)/\text{Å}$ | Ref. |
|---|---|---|---|
| $NO_2CH_2CH_2$ | 1.470 | 1.262 | 36 |
| $c\text{-}C_6H_{11}$ | 1.488 | 1.272 | 37 |
| $o\text{-}C_6H_4COOH$ | 1.460 | 1.267 | 26 |
| $2NO_2.C_6.H_{10}$ | 1.487 | 1.274 | 38 |
| $Ph\text{-}CHNO_2.CH_2$ | 1.479 | 1.269 | 38 |
| $Me_2C.NO_2$ | 1.506 | 1.251 | 39 |
| $Bu^t$ | 1.533 | 1.265 | 40 |
| $2,6\text{-}Pr_2^iC_6H_3$ | 1.467 | 1.267 | 35 |
| $c\text{-}C_3H_5$ | 1.434 | 1.268 | 41 |

above, does not alter the $\nu(N-O)/r(N-O)$ interrelationship which holds for mono-mers themselves, then it is implies that the previous uses of $\nu(N-O)$ to obtain an insight into coordination mode [6a–6k], upon which we have recently commented concerning coordination by $p$-nitrosodimethylaniline [42], are reflections of changes in $r(N-O)$ in complexes. This is the case in that the $\nu(N-O)/r(N-O)$ relationship for the coordination compounds lies well within the error limits of that for monomers. This would imply that $\sigma$-N coordination I is inevitably accompanied by a lengthening of the NO bond and $\sigma$-O coordination VII by a contraction of the bond whereas $\pi(NO)$ coordination must always be associated with a considerable lengthening of the bond. It seems to us that only the last conclusion is totally in keeping with intuitive expectation. Consequently in Table 3 and Figs. 4 and 5 we examine the $\nu(N-O)/r(N-O)$ and $r(C-N)/r(N-O)$ relationships for the coordina-tion compounds. It is evident from Fig. 4 that coordination does not alter the
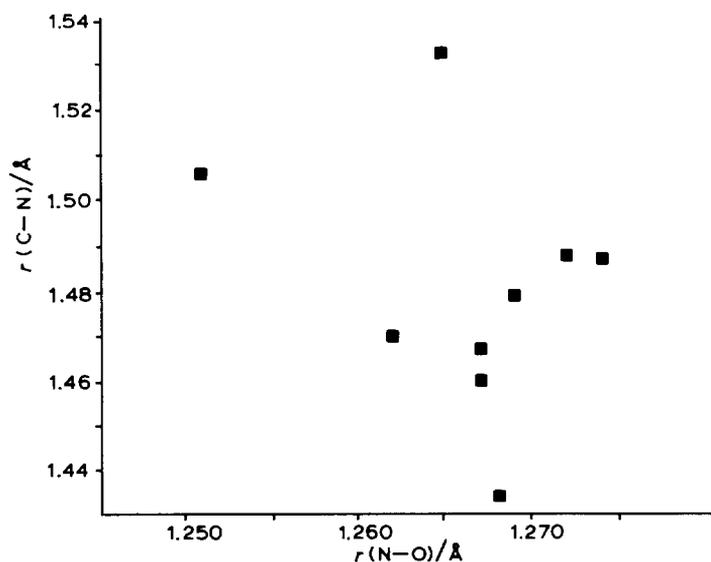


Fig. 3. Plot of C–N bond length against N–O bond length of C-nitroso dimers from X-ray data summarised in Table 2.

Table 3

Structural data for coordinated *C*-nitroso compounds

| Compound | $r$(C–N)/Å | $r$(N–O)/Å | $\nu$(N–O)/cm$^{-1}$ | Coordination mode | Ref. |
|---|---|---|---|---|---|
| CoCl$_2$(NODMA)$_2$ [a] | 1.360 | 1.267 | 1498 | I | 6d |
| Me$_2$SnCl$_2$(NODMA)$_2$ [a] | 1.398 | 1.218 | | VII | 6e |
| RhCl(NBD)(*p*-BrC$_6$H$_4$NO) [a] | 1.43 | 1.23 | 1473 | I | 6i |
| [Fe(CO)$_3$.C$_6$H$_3$.NO.Me.Cl]$_2$ | 1.43 | 1.40 | | III | 43 |
| (P$^t$Bu$_3$)$_3$.PhNO.Pd | 1.43 | 1.35 | 1039 | III | 6a |
| (CoCp)$_2$(PhNO)$_2$ | 1.423 | 1.385 | 1047 | III | 44 |
| PdCl$_2$(PhNO)$_2$ | 1.411 | 1.209 | 1490 | I | 45 |
| Pt(PPh$_3$)$_2$PhNO | 1.412 | 1.410 | 973 | II | 46 |
| (PhNO)$_3$(PMe$_3$)$_3$Pt$_2$ | 1.372 | 1.296 | | I | 47 |
| | 1.359 | 1.433 | | IV | 47 |
| | 1.459 | 1.428 | | V | 47 |
| ZnCl$_2$(NODMA)$_2$ [a] | 1.345 | 1.300 | | VII | 6g |
| C$_5$Me$_5$.Ru.Ph.PPhMe$_2$.EtNO | 1.495 | 1.258 | | I | 48 |
| W(CO)$_5$$^t$BuNO | 1.51 | 1.24 | 1415 | I | 5 |
| | 1.50 | 1.25 | 1418 | I | 41 |
| PtCl$_2$($^t$BuNO)$_2$ | 1.56 | 1.21 | 15555 | I | 6c |
| CpMn(CO)$_2$$^t$BuNO | 1.541 | 1.271 | 1325 | I | 41 |
| CpFe(CO)PPh$_3$$^t$BuNOBF$_4$ | 1.554 | 1.234 | | I | 5 |
| CpCoPPh$_3$EtNO | 1.484 | 1.282 | 1300 | I | 49 |
| Fe(TPP)$^i$PrNO.$^i$PrNH$_2$ | 1.54 | 1.26 | 1432 | I | 50 |
| | 1.55 | | | | |



| | 1.544 | 1.22 | | I | 51 |



| | 1.492 | 1.252 | 1357 | I | 52 |
| | 1.487 | 1.247 | | I | |

| C$_{15}$H$_{10}$Fe$_4$N$_2$O$_{12}$ | 1.48 | 1.45 | | VI | 58 |
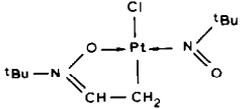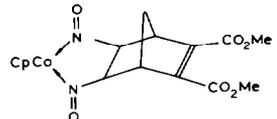
[a] NODMA = *p*-nitrosodimethylaniline; NBD = norbornadiene.

$\nu$(N–O)/$r$(N–O) relationship. On the other hand Fig. 5 demonstrates quite clearly that the compensatory relationship between $r$(C–N) and $r$(N–O) is unaltered by coordination of types I and VII, i.e. $\sigma$-N and $\sigma$-O coordination *provided that the nitroso compound is an aromatic nitroso compound*. When $\sigma$-N coordination occurs from a nitrosoalkane the CN bond appears to be lengthened by up to 0.1 Å compared with a free nitrosoalkane having the NO bond length observed in the complex. This conclusion may be phrased alternatively. The effect on the $r$(C–N)/$r$(N–O) relationship for nitrosoalkane coordination compounds is of a similar order of magnitude to that which occurs on dimerisation where there is an electronic reorganisation [53–57]. If the $r$(C–N)/$r$(N–O) relationship is based upon mutual conjugation as Talberg has suggested [3], then conjugation with the $\pi$-electrons of the ring allows this to be maintained when $\sigma$-N coordination (I) or $\sigma$-O coordination (VII) occurs. In the absence of the $\pi$ electron contribution from the ring the mutual
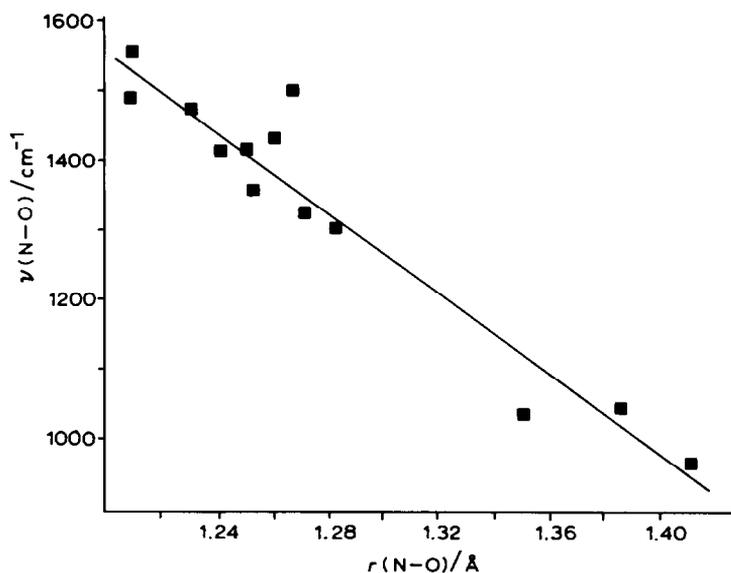
Fig. 4. Plot of N–O stretching frequency (wavenumber) against N–O bond length of coordinated C-nitroso compounds from data summarised in Table 3.

conjugation of the electrons in the C–N and N=O bonds is fundamentally altered when σ-N coordination takes place. No correlation with CNO bond angle is apparent throughout these data.
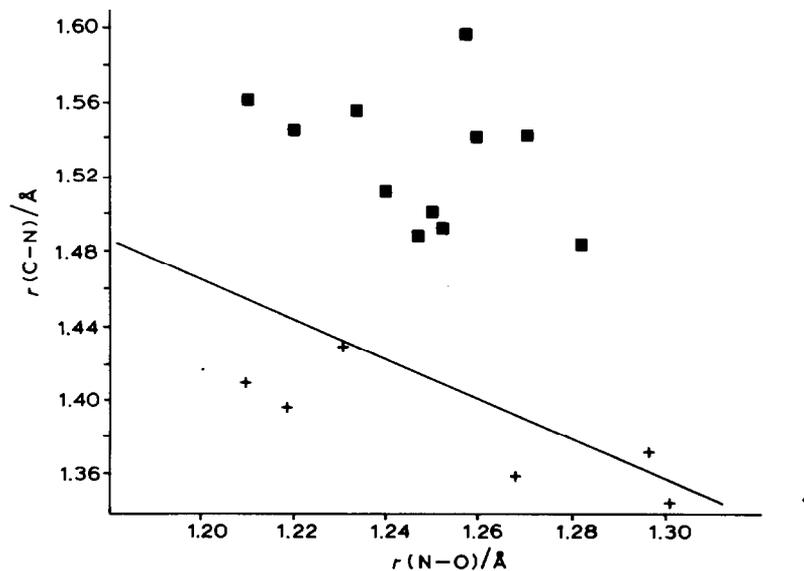


Fig. 5. Plot of C–N bond length against N–O bond length of coordinated C-nitroso compounds from X-ray data summarised in Table 3. + aromatic nitroso compounds of coordination type I and VII; ■ alkyl nitroso compounds of coordination type I; ——— line taken from Fig. 2.

It is noteworthy that the only forms of nitrosoalkane coordination that have been established are confined to type I ($\sigma$-N) with the single exception of nitrosoethane coordinated to four Fe atoms in a cluster type (VI) [58] and that type VII ($\sigma$-O) is confined to $p$-nitrosodimethylaniline as ligand and $d^{10}$ metals as coordination site. The literature data collected in Table 3 indicate that $p$-nitrosodimethylaniline does not engage in $\pi$ coordination and that nitrosobenzene can coordinate by modes I, III, IV and V.

We thus arrive at the following conclusions. (1) The use of $\Delta\nu$(N–O) for establishing coordination mode for a $C$-nitroso compound is secure for the various $\pi$-coordination modes through not for distinguishing between modes II, III, IV and VI. The loss of the $\pi$ character from the NO double bond leads to considerable lengthening of the bond and to a large drop in $\nu$(N–O). (2) When coordination occurs by modes I and VII the use of $\Delta\nu$(N–O) does not afford adequate discrimination and it is suggested that in the absence of crystal structure measurements it is safer to assume coordination by mode I except for the ligand NODMA coordinated to compounds of the $d^{10}$ metals. (3) The same $\nu$(N–O)/$r$(N–O) relationship holds within low error limits for complexes as for monomer nitroso compounds and hence measurement of $\nu$(N–O), preferably established by $^{15}$N labelling, may be used to predict $r$(N–O). (4) Coordination by nitrosoalkanes occurs by mode I except possibly for clusters and leads to pronounced lengthening of the CN bond as based upon the $r$(N–O)/$r$(C–N) relationship. This use of the lone pair in coordination by nitrosoalkanes produces a change in the $r$(N–O)/$r$(C–N) relationship which is of a similar order of magnitude to that operative when dimerisation of the nitrosoalkane occurs.

## Acknowledgements

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