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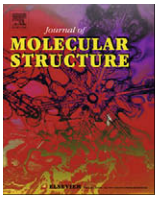

Cross-dimerization of nitrosobenzenes in solution and in solid state

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ABSTRACT

Cross-linking of nitrosobenzenes to heterodimers (azodioxides) when they are not sterically crowded with large groups in *ortho*-position was studied by NMR and FT-IR spectroscopy, X-ray crystallography as well as by cryogenic photolysis. It was found for the first time that cross-linked dimers (heterodimers) exist both in solution and in solid state and that they appear in the crystal together with homodimers. The system is used as a model for studying organic solid solution formation. The role of molecular packing in formation of a weak chemical bond is also discussed.

1. Introduction

Broad literature is available on the nitroso compound dimerization to azodioxides (Scheme 1) [1]. This monomer–dimer equilibrium in solution has been studied in detail from kinetic and thermodynamical aspects. In our recent studies, we found that azodioxides (nitroso dimers) underwent photochromic behaviour not only in solution but also in solid state [2]. Solid state dimerization of nitrosobenzenes was also proposed as a model for studying the solid state reaction mechanisms [3]. Since chemical changes in crystals include phase transformation and chemical reaction, it is not quite clear how these processes contribute to the structure of the product and how they determine the reaction mechanism. A study of the possibility of formation of cross-linked dimers (R and R′ are different, Scheme 1) by co-crystallization of monomers is in line with this investigation.

It is known from previous work that cross-linked dimers are formed if a nitroso group is sterically crowded by substituents in *ortho*-position in one of the monomer partners [4a]. No-appearance of cross-linked dimers not substituted in the *ortho*-position has been observed so far. On the other hand, we still possess no systematic knowledge about the influence of electronic and/or topochemical factors on the cross-linking selectivity, i.e., the conditions under which the monomer will combine with a partner of the same structure ($R = R'$) or with a different partner ($R \pi R'$). Some work on such selectivity has been published recently [4b].

The main problem in studies of the possible formation of cross-linked dimers is how to find the method by which these heterodimers can be unequivocally distinguished from a mixture of homodimers. Our example of the formation of heterodimers includes a combination of *p*-bromo- and *p*-nitronitrosobenzene. The mixture was obtained by crystallization of equimolar solutions of both compounds. The obtained crystals were compared with the crystals of pure homodimers (i.e., nitro–nitro or bromo–bromo azodioxides) by IR and CP MAS NMR spectroscopy, as well as by X-ray diffraction structure determination. Possible appearance of heterodimers in a crystal mixture of two homodimers represents also a model for studying solid solution formation. To confirm that cross-dimerization is not only a packing effect, but also a consequence of electronic factors involved in chemical bonding, we also conducted NMR experiments in solution.

2. Experimental

2.1. Preparation of p-nitronitrosobenzene

The compound was prepared by oxidation of *p*-nitroaniline with *oxone*® ($K_2SO_5 \times K_2SO_4 \times KHSO_4$) analogously to a method described in literature [5]. DCM solution of amine (10 mmol in 40 mL) was added in portions to a vigorously stirred mixture of DCM (10 mL) and water solution of oxone (20 mmol, 100 mL).

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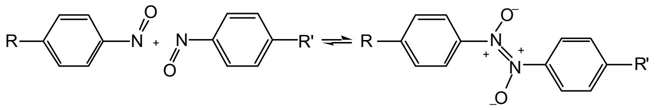


Scheme 1.

The reaction mixture was kept open to the atmosphere. At first, the solution slowly became brownish. Later, it turned green, indicating the formation of nitroso species. After approximately 5 h, the DCM layer was separated, washed twice with hydrochloric acid ($c = 1$ mol dm^{-3}, 2×20 mL) and water (20 mL). The solution was dried on anhydrous $CaCl_2$ and the DCM was separated on a rotary evaporator. We obtained a slightly brownish precipitate, the exact composition of which we have not determined. *p*-Nitronitrosobenzene was purified by sublimation under reduced pressure (10^{-2} Torr), yielding some green material on the cold finger (temperature of cooling water was around 10 °C). The colour of this material changed to yellow due to dimerization during approximately 1 h at room temperature.

2.2. Preparation of p-bromonitrosobenzene

The compound was prepared by oxidation of *p*-bromoaniline with *oxone*® ($K_2SO_5 \times K_2SO_4 \times KHSO_4$) analogously to a method described in literature [5]. Ten millimolar of *p*-bromaniline dissolved in 20 mL of DCM was added in one portion to a water solution of oxone (20 mmol in 50 mL). This resulted in formation of a white precipitate, which slowly disappeared as the mixture became greener. The mixture was vigorously stirred for 5 h. Afterwards, the layers were separated and the DCM layer was washed twice with hydrochloric acid ($c = 1$ mol dm^{-3}, 2×20 mL) and once with water (20 mL). The solution was dried on anhydrous $CaCl_2$ and DCM was separated on a rotary evaporator. We obtained a light grey solid, exact composition of which we have not determined. *p*-Bromonitrosobenzene was purified by sublimation under reduced pressure (10^{-2} Torr), yielding some green material (temperature of cooling water for the cold finger was around 10 °C). The green sublimed material turned white due to dimerization over the following several days while it was stored at −10 °C.

2.3. Preparation of ^{15}N-p-bromonitrosobenzene

^{15}N-*p*-bromoaniline was prepared according to a method described in *Vogel's Preparative Organic Chemistry* [6]. ^{15}N-aniline (0.20 mL) was converted to ^{15}N-acetanilide with acetanhydride (0.24 mL) in glacial acetic acid (0.24 mL). The reaction mixture was gently boiled for 40 min and poured into 4 mL of cold water while still hot. ^{15}N-acetanilide crystallized and was isolated with suction and washed with ~2 mL of cold water. The product was slightly pink in colour, probably due to impurities in the starting ^{15}N-aniline. We obtained 210 mg of ^{15}N-acetanilide (70%). ^{15}N-acetanilide was then dissolved in 0.70 mL of glacial acetic acid; to this solution, a solution of bromine in glacial acetic acid (0.50 mL, $c = 3.5$ mmol mL^{-1}) was added dropwise. The mixture was stirred at room temperature for ~1 h, resulting in formation of ^{15}N-*p*-bromoacetanilide precipitate. The mixture was transferred to 8 mL of water, filtered with suction and washed with ~4 mL of water, yielding 280 mg of ^{15}N-*p*-bromoacetanilide (84%).

^{15}N-*p*-bromoacetanilide was then dissolved in 0.70 mL of boiling EtOH; to this solution, 0.33 mL of concentrated hydrochloric acid was added dropwise. The mixture was heated under reflux for 50 min. Afterwards, 4 mL of water was added and the mixture was evaporated (removal of EtOH and EtOAc) until ~1 mL remained. The obtained solution was then diluted with water and 10% NaOH (aq) was added until just alkaline. The resulting precipitate of ^{15}N-*p*-bromoaniline was filtered with suction and washed with a small amount of cold water. We obtained 110 mg of ^{15}N-*p*-bromoaniline (49%).

^{15}N-*p*-bromoaniline was oxidized to a nitroso compound according to the method described above and purified by sublimation. We obtained 90 mg of ^{15}N-*p*-bromonitrosobenzene (74%) after sublimation.

2.4. Preparation of crystals of the heterodimer and two homodimers

Twenty milligrams of ^{15}N-*p*-bromonitrosobenzene and 16.4 mg of *p*-nitronitrosobenzene were dissolved in ~1.5 mL of cold (5 °C) and dry chloroform. This resulted in a green solution, which was left to evaporate in a refrigerator at 5 °C. After three days, yellow needle-like crystals were formed.

2.5. IR spectroscopy

IR spectra were recorded on a Bruker FT-IR spectrometer under 2 cm^{-1} resolution.

2.6. NMR spectroscopy

Solid-state NMR experiments were performed on a 14.1 T Varian Unity Inova spectrometer equipped with a 3.2 mm T3 MAS probe from Varian/Chemagnetis. ^{1}H ^{13}C CPMAS spectra were recorded using a standard ramped cross-polarization pulse sequence with high-power proton decoupling during acquisition. Contact time for cross polarization was 5 ms, repetition delay was 8 s and sample rotation frequency was 10 kHz for all samples. The number of scans was 2000 for pure *p*-Br and *p*-NO_2 substances, and 8000 for a physical mixture of the above two substances and for a heterodimer. The rotors contained about 100 mg of sample. (Chemical shifts are reported relative to the position of the signal of tetramethylsilane.).

The liquid-state one- and two-dimensional ^{1}H and ^{13}C NMR spectra (600.13 MHz for ^{1}H, 150.90 MHz for ^{13}C) were measured in $CDCl_3$ at 298 and 218 K on a Bruker AV600 spectrometer equipped with 5 mm TBI and 5 mm BBO probes with z-gradients. Chemical shifts, in ppm, were referred to TMS as the internal standard. The standard ^{1}H, APT and COSY experiments were used. The FID resolution in ^{1}H NMR and ^{13}C NMR spectra was 0.29 Hz and 0.54 Hz per point, respectively. The COSY technique with a standard two $\pi/2$ pulse sequence (cosygpqf) in the pulsed field gradient mode (z-gradient) was applied using 2 k data points in F2 dimension and 512 increments in F1 dimension. The latter was subsequently zero-filled to 1024 points. Increments were obtained by four scans each, 8 kHz sweep width and pulse spacing was 1.0 s. The FID resolution was 4.1 and 16.4 Hz/point in F2 and F1 dimensions, respectively.

2.7. X-ray diffraction on single crystals

Data were collected by a series of omega scans on an Oxford Diffraction Xcalibur3 CCD X-ray diffractometer with graphite monochromated Mo$K\alpha$ radiation and were processed using the CrysAlis [7] software package. Crystal structure was solved by direct methods implemented in SHELXS-97 and refined on F^2 by full matrix least squares using SHELXL-97 [8]. Both programs were used as part of the WinGX [9] software package.

Hydrogen atoms were placed in their geometrically calculated position with the C–H bond distance of 0.93 Å and with U_{iso}(H) = $1.2U_{eq}$(C). ORTEP3 [10] was used for graphical presentation of the molecular structure. We performed two data collections of the co-crystal with crystals from different batches. One set of data was collected at room temperature and the other at 100 K. No differences in crystal structures were observed except those which can be attributed to a change in temperature.

*Crystal structure of the crystal of **1*** was solved in the $P2_1/c$ space group with two molecules in one unit cell and half a molecule in the asymmetric unit. Cell dimensions at 100 K are $a = 13.010(3)$ Å, $b = 3.6827(7)$ Å, $c = 12.993(3)$ Å, $\beta = 103.87(2)°$. Cell dimensions at 293 K are $a = 13.018(4)$ Å, $b = 3.760(1)$ Å, $c = 13.168(4)$ Å, $\beta = 104.17(3)°$.

Crystal structure of dimeric p-nitronitrosobenzene was solved in the $Pbc2_1$ space group with four molecules in one unit cell. Cell dimensions at 150 K are $a = 3.664(1)$ Å, $b = 14.598(4)$ Å, $c = 22.949(10)$ Å.

3. Results and discussion

3.1. NMR spectroscopy: cross-dimerization in solution

In order to observe the possible formation of a heterodimer in solution, we performed NMR experiments in chloroform-d$_5$ at ambient (298 K) and lowered temperature (218 K). While monomers were found to be dominant species at ambient temperature, decrease of temperature caused dimerization (as shown in the COSY spectra of *p*-nitronitrosobenzene, Fig. 1a and b).

At both temperatures, the rotation of the nitroso group is free and the two *ortho*- and *meta*-protons are indistinguishable. Restricted rotation leading to distinct chemical shifts of all four

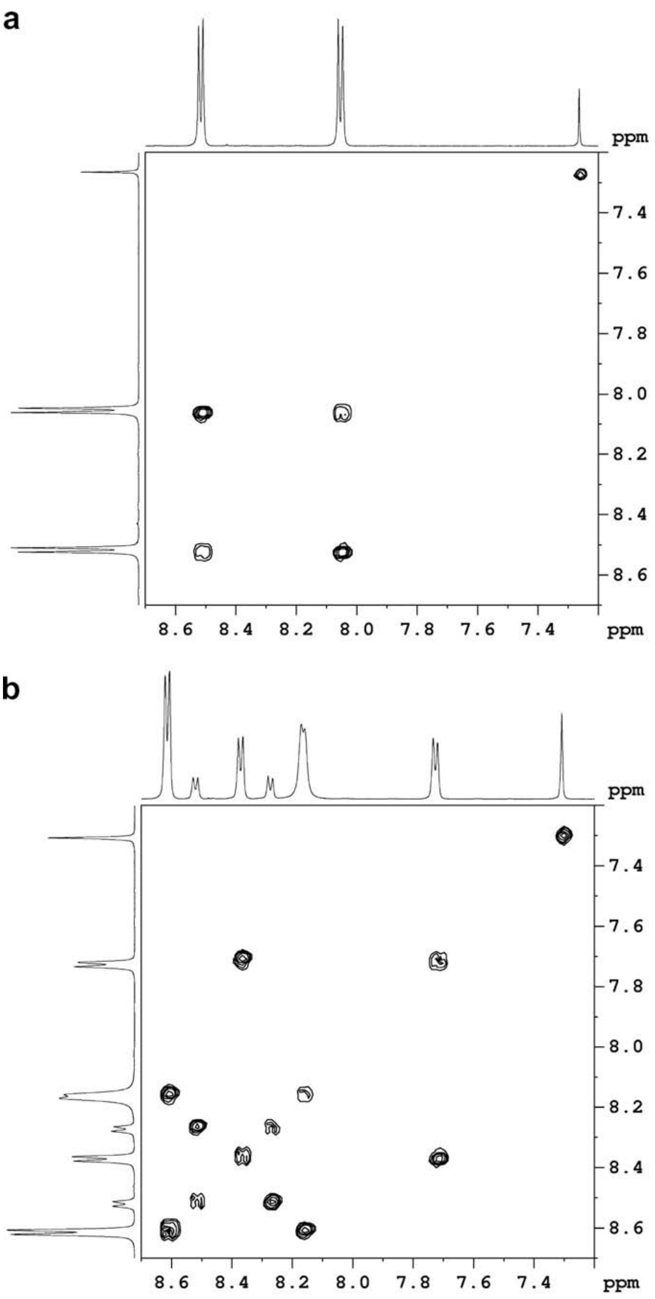


Fig. 1. (a) ^{1}H NMR COSY spectrum of *p*-nitronitrosobenzene in chloroform at 298 K. (b) ^{1}H NMR COSY spectrum of *p*-nitronitrosobenzene in chloroform at 218 K.

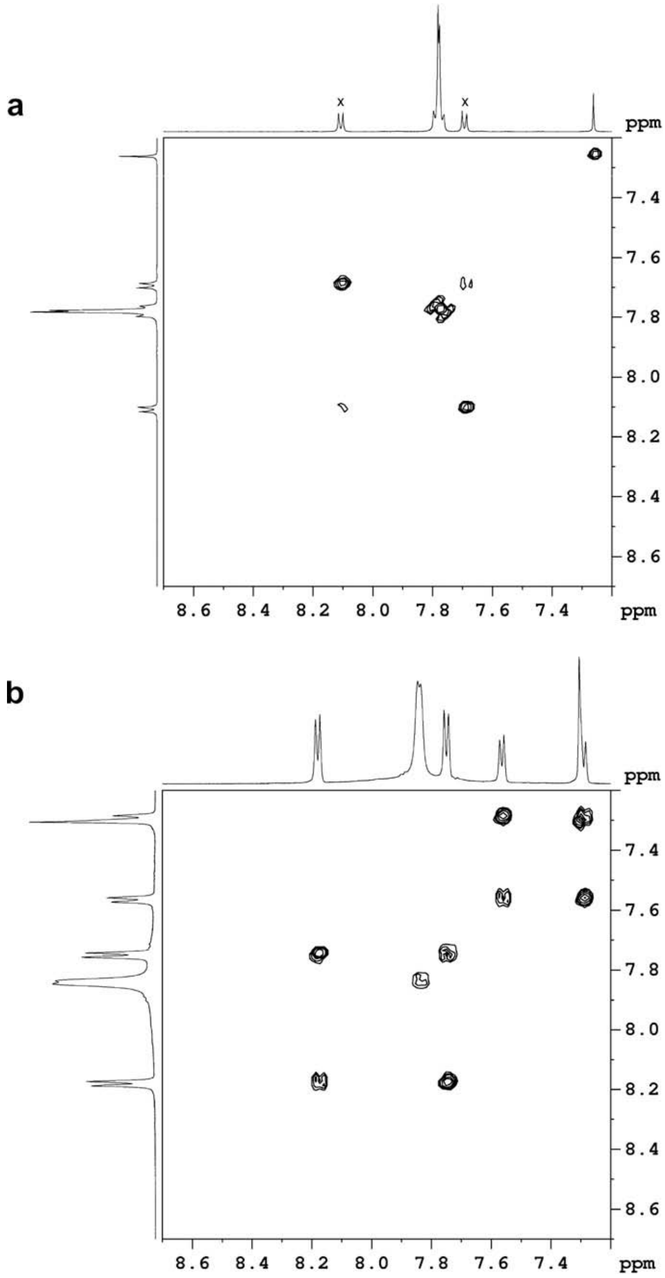


Fig. 2. (a) ^{1}H NMR COSY spectrum of *p*-bromonitrosobenzene in chloroform at 298 K. Impurity (*p*-bromonitrobenzene) is labelled by X. (b) ^{1}H NMR COSY spectrum of *p*-bromonitrosobenzene in chloroform at 218 K.

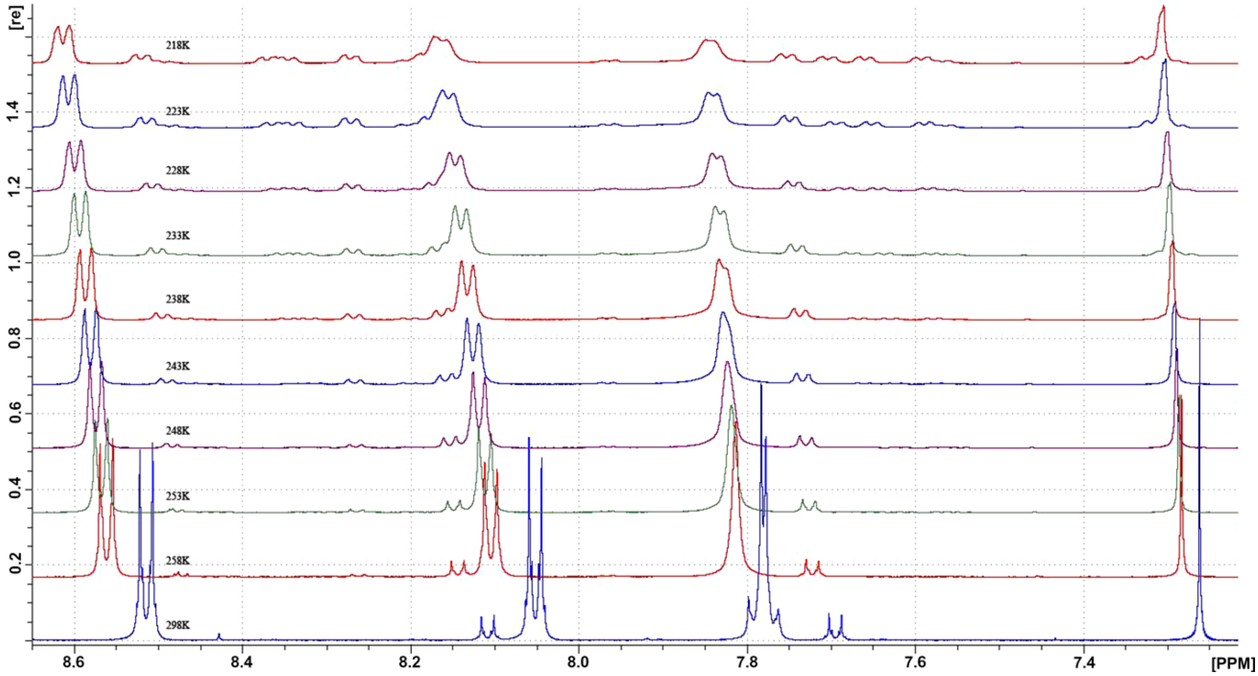


Fig. 3. ^{1}H NMR spectrum of the mixture of *p*-bromo- and *p*-nitronitrosobenzene in chloroform at different temperatures.

aromatic protons is expected at a temperature as low as −100 °C [11]. Fig. 1b displays two new sets of coupled protons for *p*-nitronitrosobenzene assigned to homodimers (*cis*- and *trans*-) that are formed by cooling. These signals disappear after warming to room temperature. Based on previous studies [11,12], we tentatively assigned the more abundant homodimer to the *cis*-form. A similar observation was also made for *p*-bromonitrosobenzene (Fig. 2a and b).

After dissolving equimolar amounts of *p*-bromo- and *p*-nitronitrosobenzene in chloroform-d$_1$, the solution was cooled to 218 K and NMR spectra were recorded and analyzed. It is clearly seen in Fig. 3 that, in addition to proton resonances assigned to monomers and homodimers, new signals appeared, which can be attributed to cross-linking products (heterodimers).

The COSY spectrum (Fig. 4) exhibited all the expected connectivities for each form. In case of a heterodimer, two phenyl groups

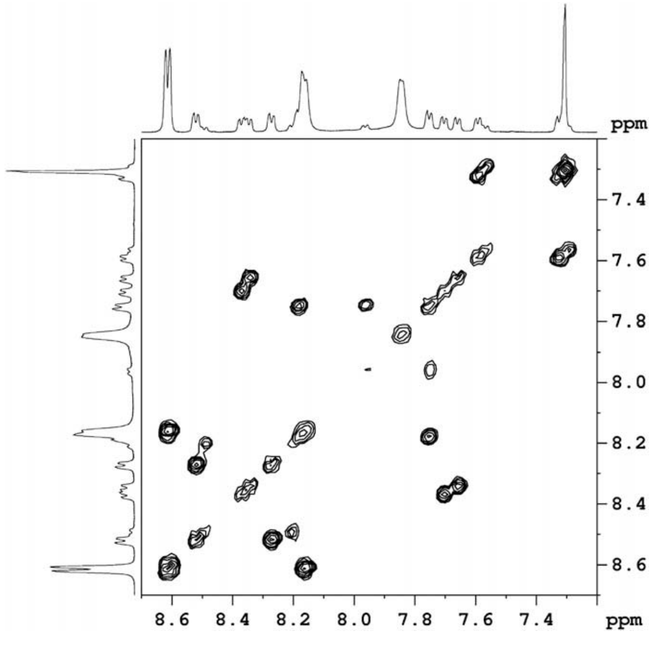
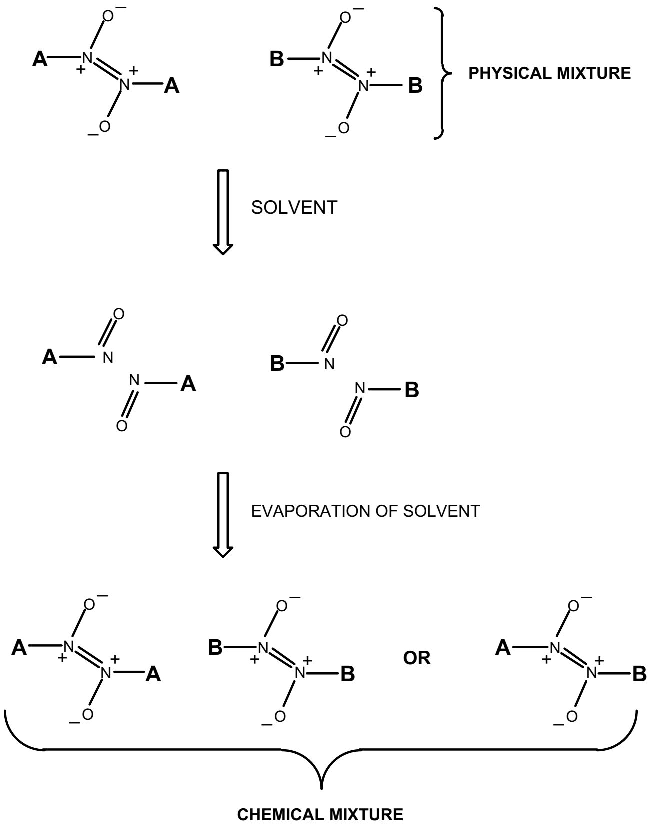


Fig. 4. ^{1}H NMR COSY spectrum of the mixture in chloroform at 218 K.



Scheme 2.

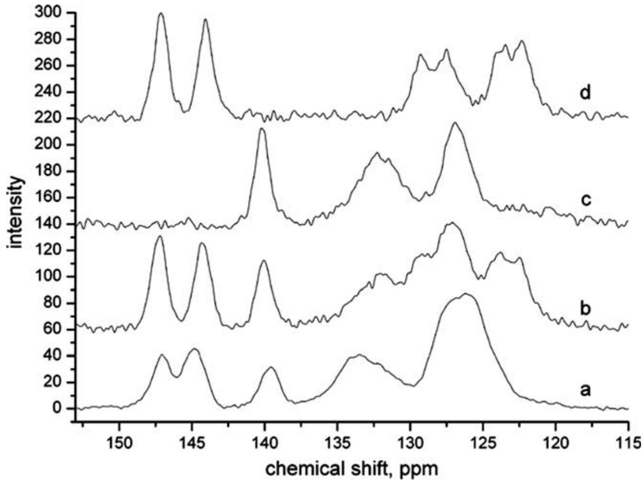


Fig. 5. ^{13}C CP MAS NMR spectra of the *chemical mixture* (a) *physical mixture* (b), pure *p*-bromonitrosobenzene dimer (c), and pure *p*-nitronitrosobenzene dimer (d).

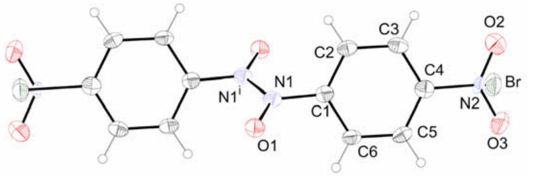


Fig. 6. ORTEP perspective of the averaged "molecule" obtained by crystallization from solution of equimolar amounts of *p*-bromo- and *p*-nitronitrosobenzene. [$i = -x, -y, -z$]. Occupancies of the bromine atom and the nitro group are 0.5.

are not equivalent, which should give rise to two different sets of coupled protons for each isomer (*cis*- and *trans*-). A close inspection of the proton spectrum and COSY cross-peaks revealed that one isomer (probably *cis*-) of the heterodimer is more populated than the other. Further experiments are needed to resolve the details of this issue.

3.2. NMR spectroscopy: cross-dimerization in solid state

CP MAS spectra of pure *p*-bromo- and *p*-nitronitrosobenzene and two different kinds of their mixtures were recorded and compared. The first mixture is called a *physical mixture*, and it consists of equimolar amounts of crystallized homodimers. The second is a *chemical mixture*, obtained by dissolving the physical mixture and evaporating the solvent. Owing to the fact that only monomers are present in solution, after evaporation of the solvent they can recombine with each other either by forming the same mixture of homodimers or by cross-linking into heterodimers (Scheme 2). If the spectra of such physical and chemical mixtures are identical or very similar, this could be an indication that only homodimers were present (**A–A** or **B–B** combinations in Scheme 2). In contrast, if spectra of a chemical mixture differ from those of a physical mixture, we can assume formation of heterodimers (**A–B** combination in Scheme 2), or appearance of new polymorphs or crystal phases, such as mixed crystals of **A–A** and **B–B** forms.

It should be pointed out here that, besides co-crystals, the resultant solid chemical mixture can also be a solid solution. The difference is that a co-crystal has three-dimensional periodicity whereas a solid solution does not. Solid solution formation arises from similarities of supramolecular synthons occurring in the crystal when synthons are formed with different chemical species. It is an advantage of solid solutions, as opposed to co-crystals, that their composition, and thus their properties, can be varied within a certain range [7a]. Therefore, this gives rise to five potential new products (physical mixture excluded), two co-crystals, two solid solutions and the disordered structure of pure heterodimer.

The solid-state NMR spectra of pure homodimers as well as of chemical and physical mixtures are shown in Fig. 5. It is evident that the *physical mixture* of homodimer crystals (spectrum *b*) is merely a sum of the spectra of pure substances (*p*-bromonitrosobenzene/spectrum *c*/and *p*-nitronitrosobenzene/spectrum *d*/). However, there are significant differences that distinguish the two mixtures. Namely, the spectrum of the *chemical mixture* (**1**) (spectrum a) exhibits a new signal at 133 ppm, whereas the signals at 122 and 123 ppm observed for the physical mixture are absent. Although the solid-state NMR spectra also reflect the molecular arrangement in the crystal, the difference in spectra *a* and *b* could be an indication of the presence of cross-linked dimers.

Crystal structure of **1** was refined in a centrosymmetric space group ($P2_1/c$) with half a "molecule" in the asymmetric unit and two "molecules" in one unit cell. The "molecular" structure obtained by X-ray analysis is given in Fig. 6. The molecule(s) lies on an inversion centre bisecting the N–N bond. This site symmetry on which the molecule lies in the structure is incompatible with the symmetry of the point group of the heterodimer molecule but this does not exclude the possibility of its presence in the crystal because the averaged electron density of the heterodimer molecules having two orientations (related by inversion, for instance) may fit the symmetry of the $P2_1/c$ space group.

When looking at the crystal structure of **1**, it is evident that it is analogous to the structure of *p*-bromonitrosobenzene dimer (Fig. 7). This structure was discussed previously [3b]. Each "molecule" forms four C–H$^m\cdots$O contacts (H^m is the hydrogen atom in *meta* position to the NO group) with four neighbouring molecules forming a layer parallel to the (a, b) plane. Structural analogy can be explained on the basis of similarity of Br$\cdots$Br and Br$\cdots$NO$_2$ interactions as supramolecular synthons [13].

The main question, whether we have really obtained the heterodimer structure of the molecule with the nitro group on one side and the bromine substituent on the other side, still remains unanswered. Namely, because of the orientational disorder, it is difficult to decide whether the molecule is an expected heterodimer or merely a disordered mixture of homodimers.

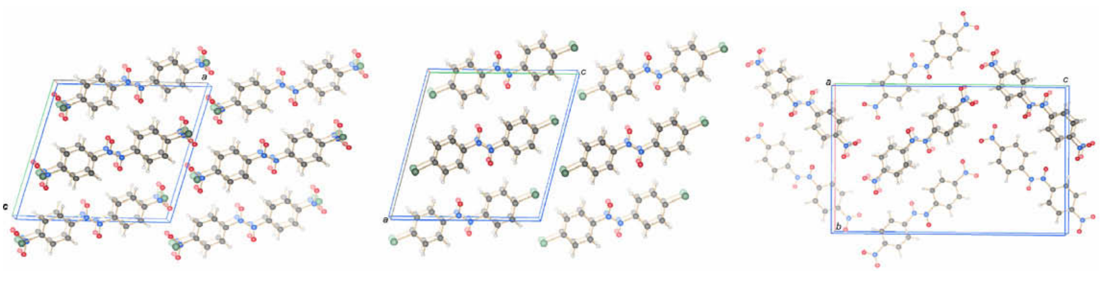


Fig. 7. Packing in **1** (left), in *p*-bromonitrosobenzene homodimer (middle) and *p*-nitronitrosobenzene homodimer (right).

3.3. FT-IR spectroscopy

The IR signal in the $1260\,\mathrm{cm}^{-1}$ spectral region, assigned to ON=NO as. stretching, can be used for characterization of the azodioxide dimeric structure. We measured its spectral position in dimers obtained from pure *p*-bromonitrosobenzene, pure *p*-nitronitrosobenzene, and their chemical mixture **1** (Fig. 8). It is interesting that this absorbance obtained from the dimer crystallized from a chemical mixture of different nitrosobenzenes (**1**) appears at the lowest frequency ($1257\,\mathrm{cm}^{-1}$). If it were just a mixture of homodimers, we would expect its appearance between the signals of pure homodimers, i.e., between 1258 and $1261\,\mathrm{cm}^{-1}$. Such indication of the existence of cross-linked azodioxide is further supported by the spectra of ^{15}N labelled compounds (Fig. 9). Namely, we prepared the ^{15}N *p*-bromonitrosobenzene and mixed it with the ^{14}N *p*-nitronitrosobenzene. If the cross-linked azodioxide appears, it must have the ^{14}N = ^{15}N signal (^{14}N = ^{14}N peak belongs to *p*-nitronitrosobenzene homodimer, and ^{15}N = ^{15}N signal is assigned to *p*-bromonitrosobenzene). As it is evident from Fig. 9, all three peaks appear in the spectrum. These findings strongly support the conclusion that by mixing equimolar amounts of *p*-bromo- and *p*-nitronitrosobenzene, all three species (two homodimers and a heterodimer) are produced, but the cross-linked molecule is dominant.

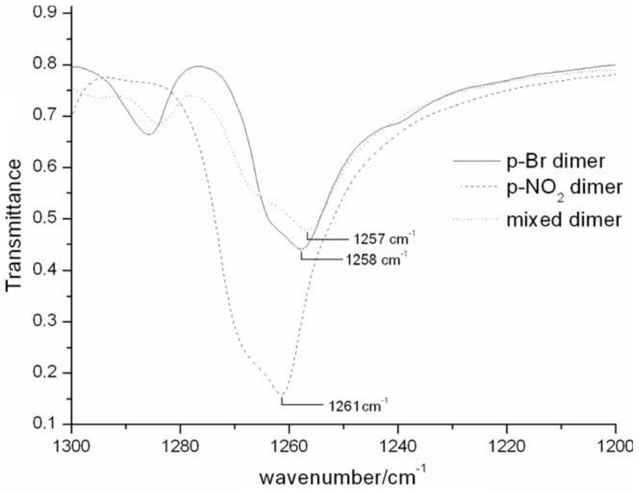


Fig. 8. FT-IR spectra of the *chemical mixture* (mixed dimer), pure *p*-bromonitrosobenzene dimer, and pure *p*-nitronitrosobenzene dimer.

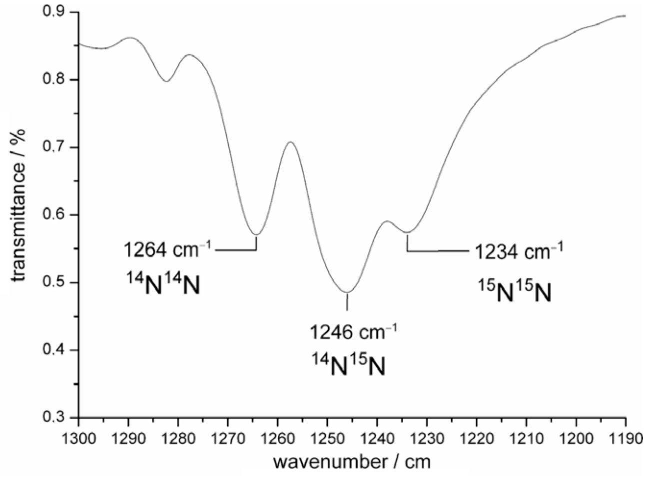


Fig. 9. FT-IR spectra of the *chemical mixture* obtained after mixing equimolar amounts of pure ^{15}N labelled *p*-bromonitrosobenzene and pure ^{14}N *p*-nitronitrosobenzene.

3.4. Co-crystal versus solid solution

In principle, if there are co-crystals of dimers that are periodically ordered, then any process of dissociation followed by recombination must disorder such a structure, and the IR spectrum before and after recombination must be different. Conversely, if there is a solid solution, it is already disordered and statistically mixed, and such recombination processes will not change the spectra. Here, we use two such recombination tests based on recombination after photolysis and after sublimation.

We know that photolysis of *p*-bromonitrosobenzene dimer yields monomers below 170 K [2a] that recombine upon warming. While photolysis is a single-crystal-to-single-crystal reaction, recombination is not, and it yields polycrystalline homodimers. The structure of photolyzed *p*-bromonitrosobenzene is such that recombination occurs with either of the two neighbouring monomer molecules. If there is a solid solution of two homodimers, then we may expect that recombination would not proceed selectively and would yield at least some amount of heterodimer. However, it is evident from Fig. 10 that the IR spectra of the *chemical mixture* before the cryogenic photolysis with the spectra obtained after photolysis followed by warming above 170 K are identical. Accordingly, dissociation and recombination in the solid state yield the same composition of homo- and/or heterodimers. If the reactant were a mixture of homo-co-crystals, and the product after recombination were a solid solution of homo and hetero dimers, then the position of the ONNO as. stretching peak at $1257\,\mathrm{cm}^{-1}$ would change its position. On the other side, if the starting mixture is *already* a solid solution, then the product mixture must be the same, and the spectrum will remain the same, as this is the case in our experiment.

A similar recombination test can be done also by sublimation. In general, sublimation of nitrosobenzene dimers yields monomer molecules that crystallize as metastable crystals on the cold finger of the sublimator. Warming to room temperature induces redimerization in the solid state [11]. When the equimolar mixture of two homodimers is sublimed and redimerized, the IR spectrum is identical to the spectrum obtained by crystallization of the same mixture from solution. If it is assumed that dimerization in these two experiments proceeded nonselectively, they strongly support the

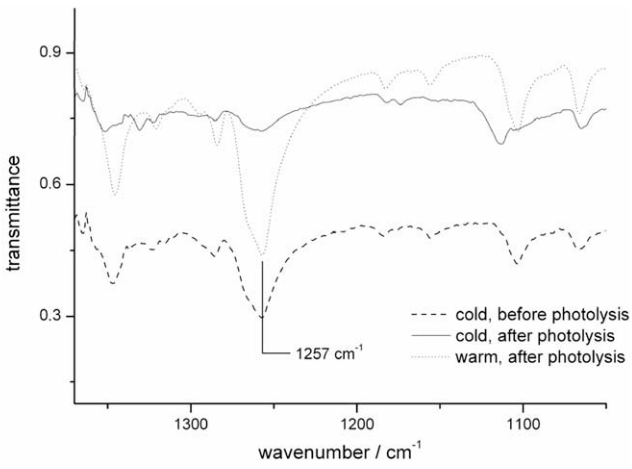


Fig. 10. IR spectra of the photolysis experiment of **1**. The dashed spectrum is displaced for clarity.

conclusion that there is a solid solution of a heterodimer and two homodimers.

4. Conclusions

We have shown here that by mixing *p*-bromo- and *p*-nitronitrosobenzenes, both homo- and heterodimers may be formed in the solid state and in solution. The appearance of cross-linked heterodimers of nitrosobenzenes sterically not crowded with *ortho*-substituents is in solid state governed by crystal packing. Since the crystal consists of all three combinations of nitroso molecules, two homodimers and one cross-linked dimer, the formation of a dimer is not a selective process, especially if one of the homodimers and the heterodimer are isomorphic by molecular arrangement. Recombination tests also suggest that there is a solid solution of a heterodimer and two homodimers. The predominant form in solution, at ambient temperature, is a monomer while a mixture of monomers, homodimers and heterodimers exists at lowered temperature. The appearance of cross-linked dimers in solution demonstrates that cross-linking is governed not only by crystal packing but also by chemical bonding.

Acknowledgements

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Appendix A. Supplementary data

Supplementary data associated with this article can be found, in the online version, at doi:10.1016/j.molstruc.2008.07.035.

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