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## **FINAL TECHNICAL REPORT**

**Shock-Induced Chemical Changes in Organic Compounds**

**Dispersed in Geologic Media**

**Submitted to  
Air Force Office of Scientific Research**

**Professor Y. M. Gupta  
Professor R. Mahalingam**

**Shock Dynamics Center  
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**July 1997**

## **Project Summary**

Over the past two decades, considerable effort has been expended to cleaning up contaminants at hazardous waste sites. Yet, to this date, thousands of sites remain to be cleaned up. The difficulty, in part, is due to the high cost and slow rate of the remediation process, through currently existing technologies. Thus, a need has been created for new in-situ approaches capable of treating large tracts of contaminated soils in a rapid time frame.

In order to examine the potential of using shock waves for promoting chemical changes in organic contaminants dispersed in geologic media, as a prelude to the development of a novel in-situ remediation technology, laboratory experiments were carried out on synthetic soil matrices containing controlled amounts of chemical impurities. Samples were subjected to peak stresses in the gigapascal range in the Shock Dynamics Center's specially designed gas gun facilities, and recovered. Chemical analyses of the recovered shocked samples through various spectroscopic techniques showed significant amounts of chemical conversion.

More specifically, in research investigations spread over a 12-month period, six organic compounds, including some examples of typical soil contaminants (acrylamide (AA), benzonitrile (BN), diphenylacetylene (DPA), nitrobenzene (NB), phenylacetylene (PA), and tetrachloroethylene (TCE)) were studied. These compounds were individually dispersed in Ottawa sand at an upper-limit concentration of 10 wt. % and subjected to shock pulses of approximately 10 GPa in amplitude and 2-3  $\mu$ s in time duration. The shock treated samples were recovered and analyzed. It was concluded that under the chosen shock conditions, AA, TCE, and PA underwent shock-induced polymerization while NB underwent decomposition. In addition, based on the intermediates and products formed, it was possible to formulate mechanistic pathways leading to their formation. It was also concluded that, in the case of BN and DPA, more research was needed in order to bring about any measurable chemical conversion.

These results demonstrate that chemical changes could be induced in organic compounds dispersed in geologic media as a function of shock wave parameters and that the potential exists for the scientific applicability of such shock waves in remediating contaminated soil in-situ.

## **Project Objectives**

The stated objectives of the research were:

- 1) to examine whether organic compounds dispersed in representative soil media could be modified using large amplitude shock waves;
- 2) to identify the product(s) formed in the shock reactions, and,
- 3) to postulate pathways through which these modifications occur.

The long term objectives of the research tying into the above immediate objectives were to understand the fundamental mechanisms governing shock induced chemical decomposition, polymerization, and catalytic activity in heterogeneous geologic media by conducting well-defined shock wave experiments on carefully characterized samples.

The above objectives were pursued by conducting planar impact experiments on a number of potential organic contaminants dispersed in a representative soil. The experiments were carried out in the 2.5 inch gas-gun facility equipped with a specialized impact fixture. At the end of each experiment, the samples were recovered for post-shock chemical analyses.

This report summarizes the experimental results to-date, allowing one to draw definitive conclusions as to whether shock-induced reactions are observed in the organic/soil mixtures studied. The possible reaction pathways for the shock reactions will be discussed in selected cases, based on the types of products formed. This summary report is expected to be a record of the status to-date of work in the successful application of shock wave transformation in chemical compounds in admixture with a representative geologic medium. Full details of the research work carried out on this project are available elsewhere [1].

## **Background**

Shock waves have been used for driving a variety of chemical reactions in both organic and inorganic compounds [2]. For example, the oldest known shock-induced chemical reaction is detonation in high explosives [3]. Using high explosives or high speed impacts, samples can be subjected very rapidly to extremely high pressures, temperatures, and shear deformations. Resulting phenomena that have been examined to date include phase transformations, polymerization and crosslinking of organic materials, powder compaction, solid state reactions in intermetallics and ceramics, and decomposition of complex molecules into simpler ones. A good understanding of the fundamental atomic / molecular mechanisms controlling these phenomena does not exist at the present time. This, however, should not preclude additional investigations not only on pure components but also on components in mixture, when the potential exists for some immediate payoffs. For example, where the component is in mixture with soil-type materials, the presence of porosity and saturation is expected to enhance shock induced chemical changes due to the

inhomogeneities present. Within the framework of a one-year study, the effort would then focus on shock modification of organic chemicals in admixture with a substrate such as an aluminosilicate, i.e., with the chemicals dispersed in an heterogeneous medium. The potential payoff from this research is expected to be in the application of the results, if successful, to the long-term problem of in-situ soil remediation.

### **Experimental Details**

**Materials and sample preparation:** Six organic compounds (AA, BN, DPA, NB, PA and, TCE) were selected because they are easy to handle, have been previously studied in a neat form under shock conditions, and several of them are potential environmental contaminants. All chemicals were purchased from Aldrich and used as received (AA, electrophoresis grade; BN, HPLC grade; DPA, 98 %; NB, 98 %; PA, 98 %; and TCE, HPLC grade). Ottawa sand, a naturally occurring  $\text{SiO}_2$ , was selected as the representative soil because sand is a major constituent of soils encountered in most waste sites. The Ottawa sand used in this work had a specific gravity of  $2.63 \text{ g cm}^{-3}$  and a particle size range of  $145 \mu\text{m} - 254 \mu\text{m}$ . It was thoroughly washed first with concentrated sulfuric acid and then with distilled water until the washings were neutral. Mixtures of AA or DPA, which are solids at room temperature, were prepared by mixing the organic compound with either NaCl or Ottawa sand in chloroform to form a slurry. After subsequent complete evaporation of the solvent, a well dispersed mixture of AA or DPA within the substrates was obtained. Mixtures of PA, NB, BN or TCE, which are liquids at room temperature, were prepared by mechanically mixing the organic compound with the substrates in a clean vial. The concentration of all mixtures thus prepared varied from 1 wt.% to 10 wt.%. Initial screening shock experiments were performed on admixtures of the organic compounds in sodium chloride (NaCl). This simplified the subsequent chemical analysis procedure as described below, because NaCl is transparent to IR and UV light.

**Shock experiments:** The shock was created by a flat copper impactor mounted on a projectile impacting on a copper cell filled with the sample. A schematic of the experimental setup is shown in Fig. 1. The impactor plate (3.18 cm diameter and 0.3 cm thick) and the cylindrical copper cell (3.18 cm diameter and 1.08 cm thick) used in this work are shown as insets in Fig. 1. The sample cavity has a volume of about  $0.25 \text{ cm}^3$ . For each shock experiment, two identical cells were loaded, each with a resulting sample bed void fraction of approximately 0.2 by volume. One of the cells was used for the actual shock experiment while the other served as a control. All results presented in this report were conducted at an impact velocity of approximately  $0.5 \text{ km s}^{-1}$ . This delivers a pressure pulse of approximately 10 GPa and an estimated pulse duration of 2-3  $\mu\text{s}$ . After the shock loading, the samples were recovered and analyzed.

**Chemical analyses:** IR (Perkin Elmer, Spectrum 2000, with Harrick diffuse

reflectance attachment) and UV (DU 65 Beckman) spectroscopy were employed primarily to detect and quantify shock induced reactions. Since NaCl is non-interfering in both techniques, no separation of the organic components from the substrate was necessary. When Ottawa sand was used, however, the organic compound had to be separated out from the sand by solvent extraction, followed by centrifugation. The extent of reaction was estimated by comparing the maximum absorbance of the organic contaminant in the UV spectra of the control and the shocked sample. Methanol was used as the solvent for all UV analyses except for AA where water was adequate. For detailed structural analysis of the control and shocked samples, H-NMR (Bruker AMX 300), GC/MS (HP 5890 II/ 5989A), and MALDI-TOF (Matrix Assisted Laser Desorption Ionization - Time of Flight) mass spectrometry (PerSeptive Biosystems, Vestec Products) were also used.

## Results and Discussion

Table 1 summarizes the typical results for the six compounds studied. All experiments were conducted under comparable loading conditions. Two of the six compounds (BN and DPA) failed to react under the particular experimental condition selected; they will likely require more intense shock conditions.

In discussing the spectral evidence for shock-induced reactions, only representative IR spectra of the organic compound/NaCl mixture are presented here for clarity, because comparable IR spectra from the organic compound/Ottawa sand mixtures were partially obscured by the bands from the solvent used for the extraction process (mostly OH bands due to methanol or moisture).

### *Acrylamide*

There are significant spectral differences between the IR spectra of the control and shocked 1 wt.% AA/NaCl samples (see Figure 2). For instance, in the shocked sample spectrum, the intensity of the C=C band is lower than that of the C=O; the opposite is observed in the spectrum of the control sample. Also, the intensity of the sharp bands between  $1500\text{ cm}^{-1}$  to  $750\text{ cm}^{-1}$  is substantially lower in the shocked sample spectrum than it is in the control sample spectrum. Clearly, the AA was modified chemically by shock loading.

The shock product from 10 wt. % AA/Ottawa sand could not directly be examined in the IR or UV as mentioned earlier. Hence, it was extracted first into methanol and then into water. The methanol and aqueous extracts were analyzed using IR and proton nuclear magnetic resonance (NMR) measurements. While the methanol extract gave IR and NMR spectra that are similar to those for pure AA, the spectra of the aqueous extract are quite

different. The latter match well with those of a commercial linear polyacrylamide (PAA). For example, most of the IR bands from PAA (Fig. 3b) can be seen in the spectrum of the aqueous extract (Fig. 3a) as well; particularly, the C-H bands at 2866 and 2939  $\text{cm}^{-1}$ , and the  $\text{NH}_3^+$  band at 2178  $\text{cm}^{-1}$ . The NMR spectrum of the aqueous extract shows the type of aliphatic protons that are characteristic of PAA. Two major sets of multiplets are observed: one at 1.1 ppm to 1.7 ppm and another at 1.9 ppm to 2.4 ppm; they represent the  $\text{CH}_2$  and the CH protons of the polymer backbone, respectively [4]. Based on the spectral evidence observed, one can conclude that PAA is the major product in the shock-induced reaction of AA in Ottawa sand. This conclusion is consistent with the literature on shock compression of neat AA [see, for example, 5, 6].

### *Tetrachloroethylene*

In experiments with 10 wt. % TCE/NaCl mixture, at least one extra band (i.e., the sharp band at 1346  $\text{cm}^{-1}$ ) and perhaps another one at 1530  $\text{cm}^{-1}$  are observed in the IR spectrum (not presented here) of the shocked sample. The new bands were not present in the spectrum of the control sample, and are considered typical of the aromatic system, Ar C=C, stretch. In the IR spectrum of the shocked 10 wt. % TCE/Ottawa sand sample (not shown), both bands were clearly observed at comparable intensity. Clearly, TCE dispersed either in NaCl or in Ottawa sand had been chemically modified by the shock loading.

Based on a total ion chromatogram from a shocked 10 wt.% TCE/Ottawa sand sample, Table 2 summarizes all the major products and their concentrations relative to the most abundant component in the product mixture, i.e., relative abundance. Clearly, the shocked sample consists of not only unreacted TCE ( $\text{C}_2\text{Cl}_4$ ) but also a few other chlorinated shock products, as identified by matching with library mass spectra.

### *Phenylacetylene*

In the case of 10 wt. % PA/NaCl samples, the major spectral changes observed in the shocked sample spectrum, when compared to that of the control sample, include the following: (i) the intensity of the  $\text{C}\equiv\text{C-H}$  and  $\text{C}\equiv\text{C}$  bands is much lower; (ii) one of the aromatic system's stretch bands, Ar-C=C, has almost disappeared (the one at 1570  $\text{cm}^{-1}$ ); (iii) the aromatic hydrogens, Ar-H, multiplet appear to be less complex (turned into a quasi-doublet); and, most interestingly, (iv) a new broad band appears near 2800  $\text{cm}^{-1}$  to 3000  $\text{cm}^{-1}$ . This new band is characteristic of an aliphatic C-H stretch. Again, similar IR spectral features were observed in the spectrum of the PA/Ottawa sand shocked sample except for the obscuring by residual solvent bands. Thus, the PA in either NaCl or Ottawa sand had been modified by the shock waves.

As before, based on the total ion chromatogram of the shocked sample, Table 2 lists a few major products and their relative abundances. The fact that the products do not dissolve readily in a solvent suggested that they might contain high molecular weight compounds. Thus, a few MALDI-TOF mass spectra of the products were taken, thus verifying the products from PA/Ottawa sand are polymeric. The NMR spectrum of a shocked 10 wt. % PA/Ottawa sand sample confirms the presence of a complex polymer (totally symmetric polymers, however, may yield sharp NMR signals). The fact that the NMR spectrum of the products shows no vinyl proton (which, if present, as in linear polyphenylacetylene (PPA), would appear somewhere close to 6 ppm) indicates that it is a cross-linked PPA. The cross-linked nature of the polymer is mirrored in its IR spectrum as well. The IR spectrum of the linear PPA reported in the literature [e.g., see Fig. 3 in [9]] is similar to the one produced under shock loading except for the extra band between  $2800\text{ cm}^{-1}$  and  $3000\text{ cm}^{-1}$  that is characteristic of aliphatic C-H stretching. This is also consistent with the observed low solubility of the product(s).

Thus, there is little doubt that shock-induced polymerization was observed in the PA/Ottawa sand mixtures. However, how does one account for the numerous minor products as revealed by GC-MS, e.g., the di- and triphenylbenzenes? One possible explanation is that the heat generated during shock compression could have thermally transformed the initial products (linear or cross-linked PPA) into various minor hydrocarbons. In a recent report [8] on thermal isomerization of *cis*-PPA, the linear polymer was found to be thermally transformed into 1,3,5-triphenylbenzene and an unidentifiable polymer (a broad H-NMR signal between 5 ppm and 8 ppm) at temperature as low as 453 K. Equally possible is that the minor products detected in the GC-MS could be an artifact of the GC instrument, where relatively high temperatures (573 K at injection port and 523 K at column) were used.

### *Nitrobenzene*

For 10 wt. % NB/NaCl mixtures, there is a major difference between the control and shocked sample spectra. The changes, however, are seemingly due to a substantial decrease in the concentration of NB in the shocked sample. All other techniques used (UV, NMR and GC-MS) for the chemical analyses of the samples pointed to the same conclusion, i.e., the major component in the shocked samples was the unreacted NB, and that its concentration was substantially lower than that in the control samples. Since loss of NB due to evaporation is not likely (its vapor pressure is extremely low, i.e., 0.133 kPa at 317 K as opposed to 0.133 kPa at 252 K for TCE [9]) it can be concluded that NB had undergone shock-induced decomposition rather than polymerization. The decomposition products are probably volatile and could not be retained at the end of each experiment. This precludes any serious elucidation of the reaction pathways for the shock reaction. Nevertheless, from the small amount

of phenol, biphenyl and nitrobiphenyl detected in the shocked samples (see Table 2), one can still put forward a reasonable degradation pathway for NB. For instance, the products obtained here, amongst other volatile species such as NO, NO<sub>2</sub>, H<sub>2</sub>O, and C<sub>6</sub>H<sub>6</sub>, could also be observed in the pyrolysis of NB in supercritical benzene [10], where high static pressure and high temperature conditions exist (100 MPa; 563-653 K).

In summary, the feasibility of shock-induced chemical changes has been demonstrated on several organic/Ottawa sand mixtures. All organic contaminants reacted were found to undergo shock-induced polymerization, except for nitrobenzene which underwent decomposition upon the application of shock.

### **Future Directions**

From the Results and Discussion Section, it is seen that the feasibility of shock-induced chemical changes has been demonstrated on several organic/Ottawa sand mixtures. In order for the results to become a commercial reality in terms of field applications for in-situ soil remediation, there need to be pursued research along the following avenues -

1. There need to be investigated a host of potential soil contaminants, both organic and inorganic, not only as single components dispersed in single soil matrices such as Ottawa sand but also as component mixtures in synthetic soil mixtures
2. There need to develop chemical sampling and analyses techniques for recovery and identification of product intermediates, a challenge indeed, in order to establish definitive chemical conversion pathways; this would help in subsequent scale-up to field applications
3. There need to develop simulation models for quantifying the contributions of each of the shock parameters in the overall chemical conversion; this information could then be utilized in the design of field applications,

The current results and the future directions envisaged should be of interest to agencies such as EPA, DOE, DOD, and DOT

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Table 1.  
Summary results from six organic compounds

Sample Mixture	Shock-induced changes detected	Extent of reaction * (%)
1 wt. % AA/NaCl	yes	39 ± 9
10 wt.% TCE/NaCl	yes	-
10 wt.% PA/NaCl	yes	61
10 wt.% NB/NaCl	yes	47 ± 4
10 wt. % AA/Sand	yes	27 ± 18
10 wt.% TCE/Sand	yes	49 ± 18
10 wt.% PA/Sand	yes	86 ± 3
10 wt.% NB/Sand	yes	51 ± 13
5 wt.% DPA/NaCl	no	< 1
10 wt.% BN/NaCl	no	< 1

\* Averaged from a set of 2 -3 experiments per sample mixture; estimated by UV and GC-MS spectroscopy. The deviations indicated are a consequence of the limited number of experiments conducted

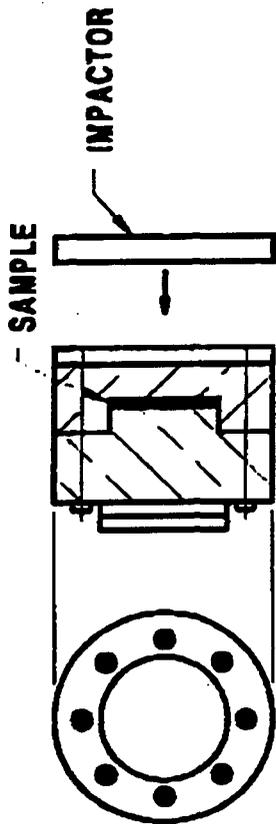
Table 2.  
Typical products from 10 wt. % TCE, PA and NB in Ottawa sand \*

Compound	Relative Abundance ‡
<i>(I) 10 wt. % TCE/Ottawa sand:</i>	
Tetrachloroethylene (C <sub>2</sub> Cl <sub>4</sub> )	100
Trideutero trichloropropenoate (C <sub>4</sub> D <sub>3</sub> Cl <sub>3</sub> O <sub>2</sub> )	31.3
Hexachloroethane (C <sub>2</sub> Cl <sub>6</sub> )	82.9
Hexachloro-1,3-butadiene (C <sub>4</sub> Cl <sub>6</sub> )	72.0
Octachloro-2-butene (C <sub>4</sub> Cl <sub>8</sub> )	47.9
Hexachlorobenzene (C <sub>6</sub> Cl <sub>6</sub> )	64.5
Octachlorostyrene (C <sub>8</sub> Cl <sub>8</sub> )	21.8
<i>(II) 10 wt. % PA/Ottawa sand:</i>	
Phenylacetylene (C <sub>8</sub> H <sub>6</sub> )	100
Styrene (C <sub>8</sub> H <sub>8</sub> )	24.4
1H-indene (C <sub>9</sub> H <sub>8</sub> )	15.3
Naphthalene (C <sub>10</sub> H <sub>8</sub> )	20.6
Biphenyl (C <sub>12</sub> H <sub>10</sub> )	37.4
4-methyl-phenanthrene (C <sub>15</sub> H <sub>12</sub> )	20.6
1-(phenylmethylene)-1H-indene (C <sub>16</sub> H <sub>12</sub> )	38.9
2-phenyl-naphthalene (C <sub>16</sub> H <sub>12</sub> )	23.7
1,3-diphenylbenzene (C <sub>18</sub> H <sub>14</sub> )	18.3
1,2,4-triphenylbenzene (C <sub>24</sub> H <sub>18</sub> )	71.8
1,3,5-triphenylbenzene (C <sub>24</sub> H <sub>18</sub> )	38.2
<i>(III) 10 wt. % NB/Ottawa sand:</i>	
Nitrobenzene (C <sub>6</sub> H <sub>5</sub> NO <sub>2</sub> )	100
Benzaldehyde (C <sub>7</sub> H <sub>6</sub> O)	< 0.5
Phenol (C <sub>6</sub> H <sub>6</sub> O)	1.4
Benzonitrile (C <sub>7</sub> H <sub>5</sub> CN)	< 0.5
Biphenyl (C <sub>12</sub> H <sub>10</sub> )	0.7
3-nitro-1,1'-biphenyl (C <sub>12</sub> H <sub>9</sub> NO <sub>2</sub> )	< 0.5

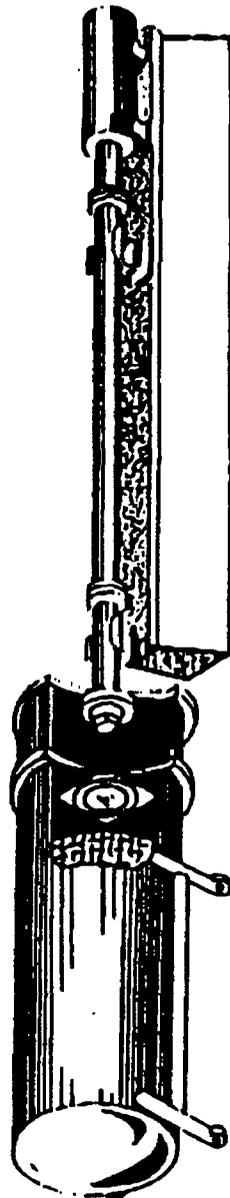
\* Products were extracted into either methanol-*d*<sub>4</sub> or chloroform-*d* and identified by GC-MS.

‡ Normalized to the most abundant component in the shocked sample extract.

Fig. 1



COPPER CELL



TYPICAL LIGHT GAS GUN FACILITY

Fig. 1 An illustration of a typical light gas gun used for shock wave experiments. The inset shows the details of the copper cell and the impactor plate

Fig 2

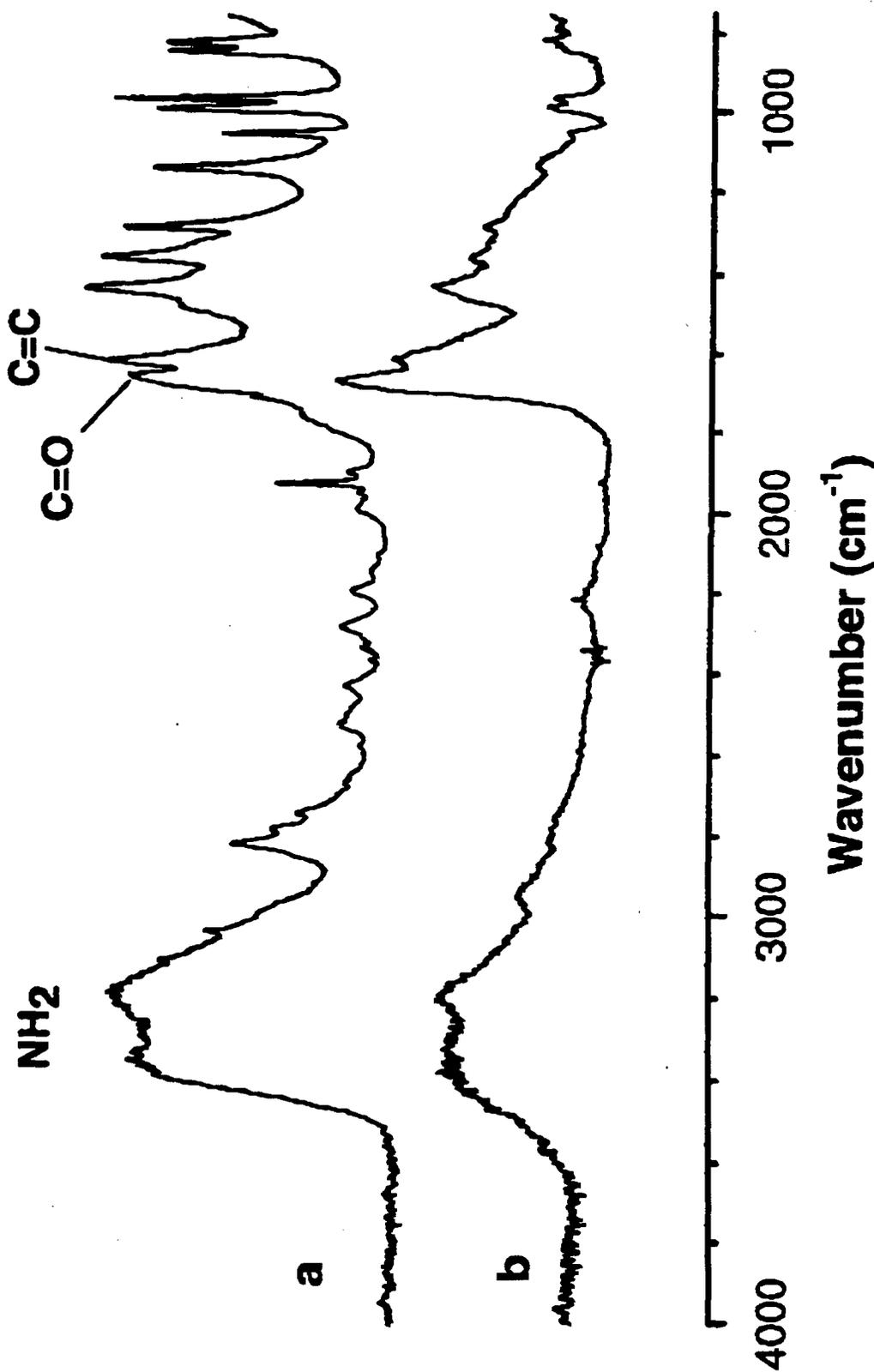


Fig. 2 FTIR spectra of a 1 wt. % acrylamide (AA) in NaCl; a) control; and, b) shocked

Fig 2

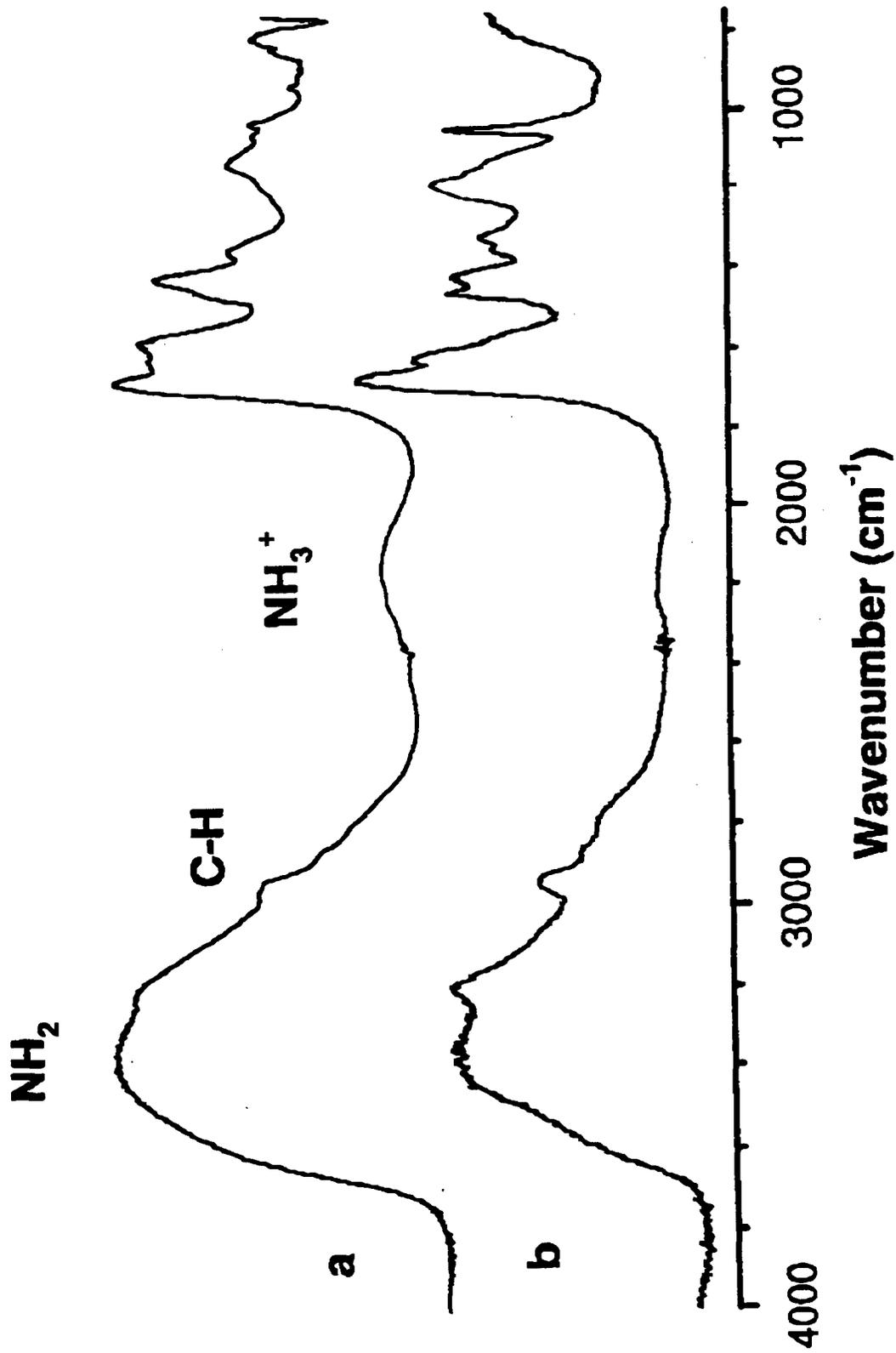


Fig. 3 FTIR spectra of (a) an aqueous extract of a 10 wt. % acrylamide/Ottawa sand shocked sample; and (b) commercial polyacrylamide