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Spectral identification of nitro compounds using a terahertz source based on an impact ionization avalanche transit-time diode

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The terahertz (THz) transmission spectra of nitro-compound layers were studied using a THz source based on an impact ionization avalanche transit-time (IMPATT) diode, as well as a photoconductive antenna. The THz spectra were recorded with a Fourier spectrometer based on a Michelson interferometer. A THz video camera with a microbolometer matrix was used as a THz radiation detector. The possibility of spectral identification of nitro compounds by using a THz source based on an IMPATT diode was experimentally demonstrated due to the presence of intense characteristic absorption bands of these substances in the range of its harmonics (0.58-1.45 THz).

Keywords: THz, spectroscopy, IMPATT, nitro compounds, identification.

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Many nitrogen-containing organic compounds have characteristic absorption bands in the terahertz (THz) frequency range. Due to this, it is possible to detect and identify such hazardous substances by using THz-spectroscopy [1–5]. Nowadays, investigations of condensed-matter THz-spectra actively employ photoconductive antennas that allow obtaining radiation in the frequency range of 0.3 to 3 THz and higher; this range contains characteristic absorption bands of a number of molecular crystals and polymers [1]. However, the necessity of using complex and bulky femtosecond lasers for exciting THz-radiation in photoconductive antennas almost fully restricts their applicability to laboratory research.

At present, researchers begin actively use compact highpower THz-sources based on an impact ionization avalanche transit-time (IMPATT) diodes [6,7]. However, the fundamental operating frequency of such sources typically does not exceed 0.6 THz. It is known that the THz radiation spectrum of sources of this type can contain, in addition to the line at the fundamental operating frequency, lines of harmonics [8,9] whose radiation power is significantly lower than that at the fundamental frequency. Taking into account the progress in developing high-power IMPATT diodes [8,10], it is possible to expect also harmonic radiation power characteristics sufficient for spectral studies in the range of at least up to 1.5 THz.

This paper is devoted to experimental investigation of the possibility of spectral studies and identification of nitro compounds with the aid of an IMPATT-diode-based THz source with the fundamental operating frequency of 0.29 THz.

Notice also that, being developed, such an approach will allow designing compact THz-scanners (inspection systems) and THz radio-vision systems capable of detecting

potentially dangerous objects not only by recording contrast THz-images, but first of all by spectral identification; high penetrability of THz-radiation through different-type dielectric barriers and absence of ionizing impacts on biological objects make this approach extremely attractive for developing THz security screening systems.

The studied samples had the form of hexogen (RDX) and trotyl (TNT) layers deposited from acetone solutions onto films of low-density polyethylene (LDPE). Solution of one of those substances was applied with an automatic pipette onto a stretched polyethylene film region 5 cm in diameter. Mean thickness of the stretched LDPE film was $10 \,\mu m$. Polyethylene was chosen as a substrate material because of its low absorption index $(< 5 \text{ cm}^{-1})$ and, in addition, because of the absence of significant spectral features in the entire frequency range under consideration [11]. As a result, samples with surface concentrations of 10 mg/cm^2 were created. Solutions of nitro compounds were prepared at the Mendeleev Russian Chemical Technical University (Moscow, Russia) and intended for certifying analytical equipment according to RF regulations. Optical microscopy examination of the prepared samples showed that the typical size of their inhomogeneities does not exceed $50 \,\mu m$.

THz transmission spectra were measured at an experimental spectral-resolution THz radio imaging setup (Fig. 1) with two THz-sources: a photoconductive antenna and IMPATT-diode-based source.

As the first THz radiation source, photoconductive antenna iPCA-21-05-1000-800-x (Batop GmbH) was used. To obtain THz pulses, the antenna was subjected to focused radiation from a femtosecond titanium-sapphire laser TiF-100ST-F12-M (Avesta Project Ltd.) with the mean radiation power of W = 2.3 W and wavelength of $\lambda = 800$ nm. The



Figure 1. Experimental setup. 1 — femtosecond laser, 2 — lens for laser radiation, 3 — photoconductive antenna, 4 — THz-lens, 5 — flat metal mirror, 6 — beam-splitting plate, 7 — stationary mirror, 8 — movable mirror, 9 — sample under study, 10 — THz video camera.

laser pulse length was $\tau = 60$ fs; the pulse repetition rate was f = 80 MHz. During the measurements, the mean power of laser radiation acting on the photoconductive antenna was continuously monitored.

Besides the photoconductive antenna, a compact IMPATT-diode-based THz source (Terasense Group, Inc.) was used; its fundamental operating frequency was 0.29 THz, while the rated mean power was 20 mW. The laser, lens for laser radiation and photoconductive antenna were excluded from the setup optical scheme (Fig. 1) and replaced with the above-mentioned source equipped with a THz-radiation attenuator.

In studying transmission spectra by using both the continuous and pulsed THz sources, it is convenient to use Fourier spectrometry. In our case, the spectra of the samples were studied using Fourier spectrometer FT-Scan (Avesta Project Ltd.) based on a Michelson interferometer (shown by the dashed line in Fig. 1).

As a radiation detector, the setup employed a Microxcam-384I-THz (INO) THz video camera based on a microbolometer matrix. The video camera was equipped with an f/1.3 lens and filter suppressing background IR light. During all the measurements, the video camera matrix was located in the rear focal plane of the lens. The samples to be studied were placed immediately in front of the video camera. THz-radiation was incident normally to the sample surfaces. The video camera was used also to monitor the radiation scattering from the studied samples [12], which appeared to be insignificant over the entire frequency range under consideration.

The study was performed in air at the temperature of 23° C and relative humidity of 30%.

Fig. 2 presents the obtained emission spectra of the IMPATT-diode-based THz- source and background signal.

The measurements were performed at the maximal spectral resolution possible for the used experimental setup. The full width at half maximum (FWHM) of each line in Fig. 2 is about 5 GHz. One can see that, besides the line at the fundamental operating frequency of 0.29 THz, the radiation spectrum of the THz-source contains also harmonic lines in the range of 0.58-1.45 THz (shown by arrows in Fig. 2); the amplitude of each of those lines is many times higher than the background signal. What is important is that this harmonic range almost fully coincides with the range of $\sim 0.8-1.5$ THz containing several known absorption bands of RDX [1].

Fig. 3, a presents the RDX sample transmission spectra obtained by using the IMPATT-diode-based THz-source (points in 2) and photoconductive antenna (points in 1).

The results show that the THz transmission spectrum of the RDX sample measured by using the photoconductive antenna contains several local minima associated with characteristic absorption bands of hexogen (shown by arrows in Fig. 3, *a*); this is in good agreement with the known RDX spectra [1]. Important is that not only in the case of the photoconductive antenna, but as well as in the case of the IMPATT-diode-based THz-source, the RDX transmission spectrum contains a feature looking as a local minimum within the intense absorption band at ~ 0.8 THz (relative to the transmittance factor at the above-mentioned line frequencies of 0.29, 0.58 and 1.16 THz). Within the measurement error, both the recorded THz-spectra are in good agreement with each other over the entire considered range.

For comparison with the RDX spectra, in Fig. 3, a there are presented TNT transmission spectra measured by using



Figure 2. Emission spectra of the IMPATT-diode-based THz-source (1) and background signal (2) (with no regard for spectral sensitivity of the THz video camera).



Figure 3. a — THz- transmission spectra of the RDX sample obtained by using an IMPATT-diode-based THz-source and photoconductive antenna (the confidence probability is 0.99). I — RDX sample (photoconductive antenna), 2 — RDX sample (IMPATT-diode-based source), 3 — pure substrate (photoconductive antenna), 4 — pure substrate (IMPATT-diode-based source). b — the same for the TNT sample. I — TNT sample (photoconductive antenna), 2 — TNT sample (IMPATT-diode-based source), 3 — pure substrate (photoconductive antenna), 2 — TNT sample (IMPATT-diode-based source), 3 — pure substrate (photoconductive antenna), 4 — pure substrate (IMPATT-diode-based source), 3 — pure substrate (photoconductive antenna), 4 — pure substrate (IMPATT-diode-based source).

the IMPATT-diode-based THz-source (points in 2) and photoconductive antenna (points in 1). Fig. 3, b shows that, regardless of the used THz-source, the TNT transmission spectra have a monotonic character free of pronounced features. This is explained by the fact that, contrary to the RDX absorption, the TNT absorption increases monotonically with frequency and has no intense bands in the range of 0.29-1.45 THz [1].

Thus, the paper shows that, in addition to the line at the fundamental operating frequency of 0.29 THz, the emission spectrum of the IMPATT-diode-based THz-source contains also harmonic lines in the range of 0.58–1.45 THz, whose power is sufficient for spectral studies. The experiments showed that RDX can be identified by its THz transmission spectrum measured with the aid of such a THz- source. The use of powerful and compact THz-sources based on IMPATT diodes will simplify the design of THz systems for spectral identification of substances and significantly extend the detection range of objects.

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Conflict of interests

The authors declare that they have no conflict of interests.

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