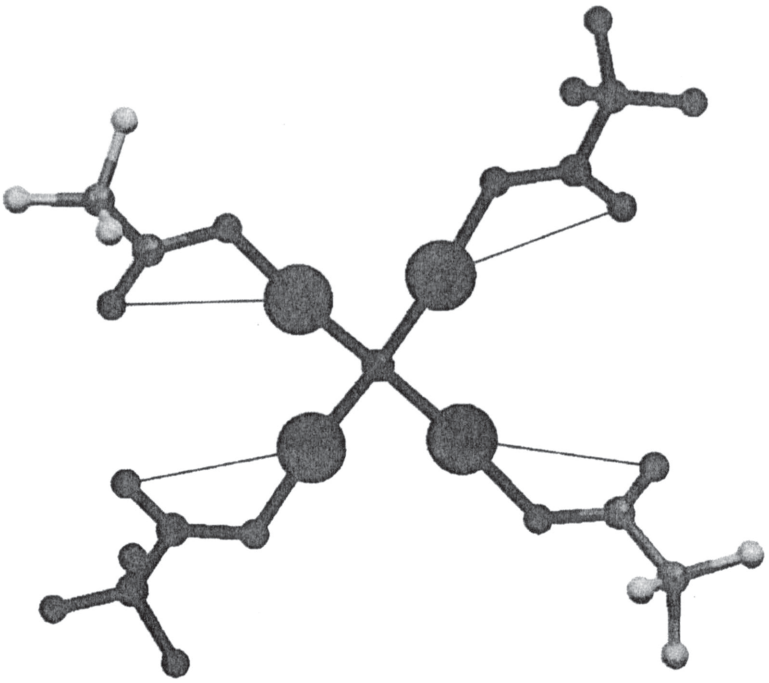
HRVATSKA AKADEMIJA ZNANOSTI I UMJETNOSTI
RAZRED ZA MATEMATIČKE, FIZIČKE I KEMIJSKE ZNANOSTI
HRVATSKA KRISTALOGRAFSKA ZAJEDNICA

KRISTALOGRAFIJA U HRVATSKOJ



Zagreb, 2013

CRYSTALLOGRAPHY IN MACEDONIA – COLLABORATION OF MACEDONIAN AND CROATIAN CRYSTALLOGRAPHERS

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Historical Overview

The first results reported by a Macedonian researcher using X-ray scattering measurements are those of Stefan Pocev of the Faculty of Technology and Metallurgy (SS Cyril and Methodius University) in Skopje, who worked (1970—1973) with professor Georg Johansson at the Royal Institute of Technology in Stockholm, Sweden. In 1973, Pocev published in *Acta Chemica Scandinavica* (Pocev & Johansson, 1973) the paper "*An X-ray Investigation of the Coordination and the Hydrolysis of the Uranium(IV) Ion in Aqueous Perchlorate Solutions*", followed by a short communication (Pocev, 1974) and another paper (Pocev *et al.* 1979) in the same journal. In 1978 Stefan Pocev completed his PhD thesis on determination of crystal structure of some mercurated oxonium and sulphonium salts at the University of Zagreb, and the results were published in the *Journal of Chemical Society, Dalton Transactions* (Kamenar *et al.* 1985).

From 1972 through 1974, Gligor Jovanovski from the Institute of Chemistry, Faculty of Natural Sciences and Mathematics (SS Cyril and Methodius University) in Skopje stayed as a postgraduate student in the X-ray Laboratory of Professor Drago Grdenić and Professor Boris Kamenar at the University of Zagreb, where he worked on determination of the crystal structure of tetrakis(trifluoroacetoxymercury)methane, the first example of a carbon atom bound to four mercury atoms in a nearly regular tetrahedral arrangement (Grdenić *et al.* 1974; Grdenić *et al.* 1982) (see Fig. 1).

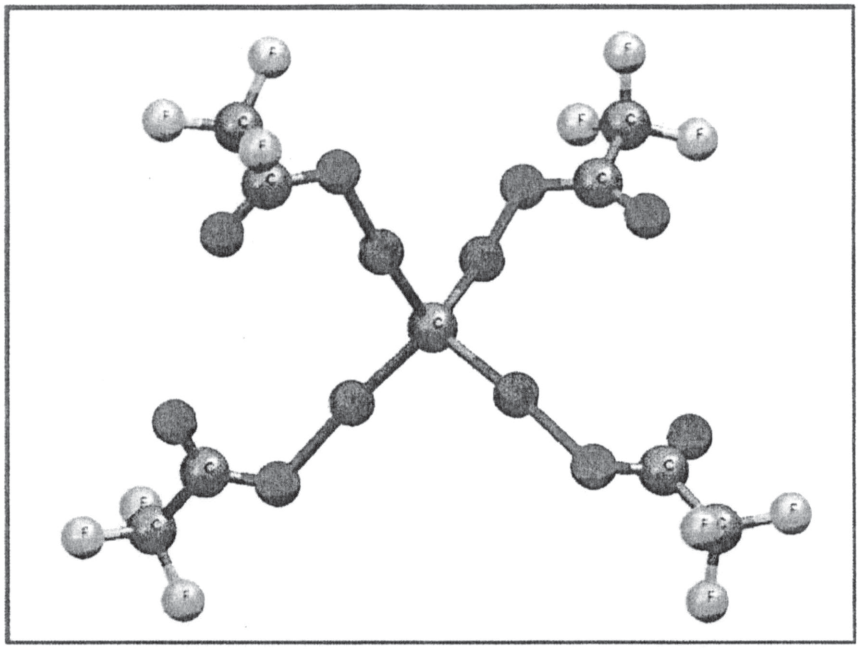

Figure 1 Crystal structure of $C(HgOCOCF_3)_4$

Later (1978—1981) Gligor Jovanovski continued the research on his PhD thesis at the same University, where he determined several crystal structures of metal saccharinate complexes (Jovanovski & Kamenar, 1982; Kamenar & Jovanovski, 1982; Kamenar *et al.*, 1982) (see Figures 2 and 3).

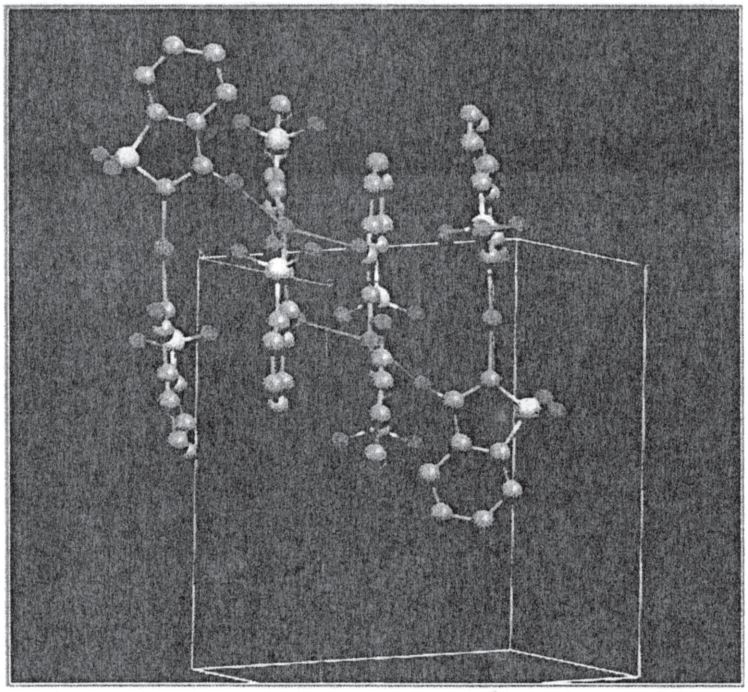

Figure 2 Crystal structure of $Hg(C_7H_4NO_3S)_2$ (C-grey; Hg-pink; S-yellow; N-blue; O-red)

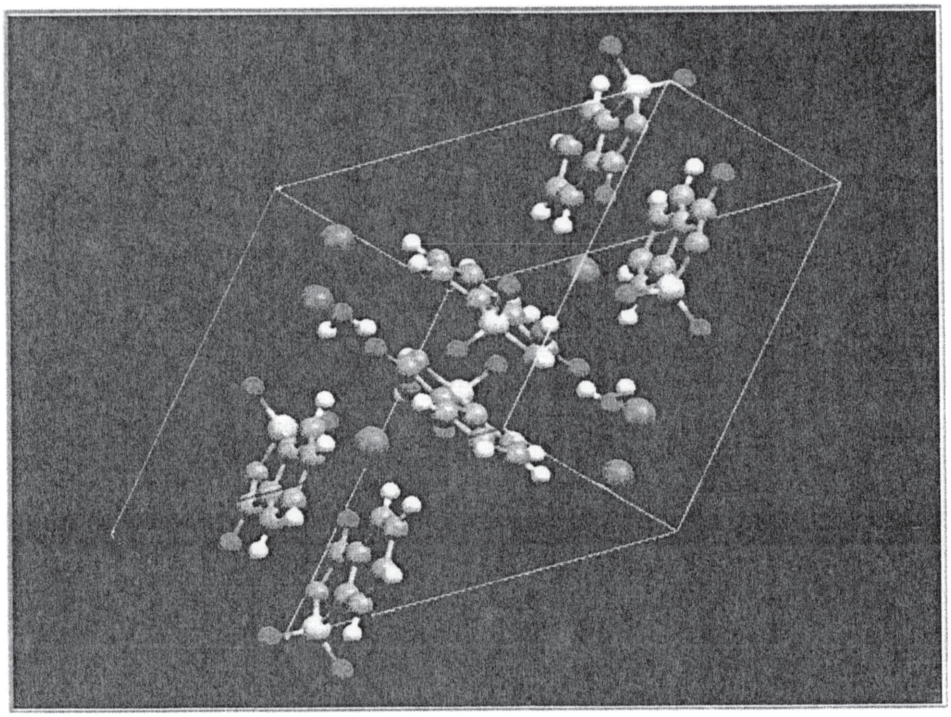

Figure 3 Crystal structure of $Na_3(C_7H_4NO_3S)_3 \cdot 2H_2O$ (C-grey; Na-violet; S-yellow; N-blue; O-red; H-white)-

After moving back to Skopje in the early 1980s, Jovanovski was the first professor to introduce a course in crystallography at the largest national University in Macedonia, the SS Cyril and Methodius University in Skopje. The course was included, as a part of the Structure of Molecules major, in the curricula at the Department of Chemistry of the Faculty of Natural Sciences and Mathematics. At that time (1975-1995), only two powder X-ray diffractometers were available in Macedonia–a *Jeol* instrument, operated by the Faculty of Natural Sciences, and a *Philips* instrument operated by the Faculty of Technology and Metallurgy. The two instruments were used for routine analyses, characterization, simple experiments and teaching.

In the following years, a small group of crystallography researchers was established at the Institute of Chemistry, including Gligor Jovanovski, Aneta Mirčeva, Orhideja Grupče, Panče Naumov, Petre Makreski and Tomče Runčevski. Sporadically, X-ray diffraction was employed for structure determination and characterization by other members of the Institute of Chemistry: Bojan Šoptrajanov, Vera Jordanovska, Vladimir Petruševski, Slobotka Aleksovska, Viktor Stefov, Gjorgji Petruševski and Sandra Dimitrovska-Lazova. Working mainly on the investigation of the structural characteristics of various sulfate and phosphate salts, they have published about 15 papers (e.g. Petruševski & Trenčevski, 1986; Petruševski, 1994; Aleksovska *et al.*, 1997; Jovanovski *et al.*, 1997; Aleksovska *et al.*, 1998; Petruševski & Aleksovska 1999; Nyborg *et al.*, 2000).

In addition, Macedonian crystallographers have also published about 30 papers related to miscellaneous studies, 6 of them being in collaboration with Croatian crystallographers (e.g. Penavić *et al.*, 1986; Kaitner *et al.*, 1992a; Kaitner *et al.*, 1992b; Jordanovska *et al.*, 2001; Naumov *et al.*, 2002a; Jovanovski *et al.*, 1987; Jovanovski *et al.*, 1993; Stefov *et al.*, 2004; Neumann *et al.* 2004; Petruševski *et al.*, 2008; Naumov *et al.*, 2010a).

Until 1989, when the country was still a part of SFR Yugoslavia, Professor Jovanovski has been a regular participant of the Annual Meetings of the Yugoslav Center of Crystallography, and he also organized one of the meetings (in 1981) in Skopje, Republic of Macedonia. Since 1996 Jovanovski has continuously participated in the Annual Croatian-Slovenian and Slovenian-Croatian Crystallographic Meetings, occasionally together with other Macedonian crystallographers (Stefan Pocev, Vera Jordanovska, Aneta Mirčeva, Orhideja Grupče, Panče Naumov, Petre Makreski and Tomče Runčevski). Very recently, a Division of Crystallography was established as part of the Society of Chemists and Technologists of Macedonia, which represents the crystallographers and structural chemists from the country.

Current Condition and Activities

Very recently (2011) three new powder X-ray diffractometers are available in Macedonia (Institute of Chemistry, Faculty of Natural Sciences and Mathematics, Skopje; Faculty of Natural and Technical Sciences, Štip and National Conservation Center, Skopje). However, owing to the nearly two decades of economic transition, none of the national or private Universities in Macedonia (a total of about ten institutions) operates modern single crystal crystallographic equipment. The results of the small group of active Macedonian crystallographers have been obtained either during their research work in foreign universities, or as a collaboration with such institutions (Jovanovski & Naumov, 2011).

Dr. Panče Naumov and Dr. Petre Makreski, both supervised in the past by Professor Jovanovski, appear as the most promising young scientists that are currently active in the field. Recently (2011) Tomče Runčevski also became active in the field of X-ray powder diffraction working on his PhD Thesis at Max Planck Institute for Solid State Research in Stuttgart, Germany, supervised by Professor Dinnebier.

The main research subjects of Macedonian crystallographers have been related to chemical crystallography (especially, metal-coordination compounds as models for biological effects of food additives), inorganic compounds (spectra-structural correlations in isomorphous and isotypic series), organic compounds with interesting electronic, optical and other properties, solid-state reactivity (photochromism, thermochromism and related reactions), organic polymorphism of pharmaceuticals, and very recently, minerals that naturally occur in Macedonia.

A work on a few projects is currently active, including the preparation of a monograph *Minerals from the Republic of Macedonia with an Introduction to Mineralogy,* which is to be published soon (in English) by the Macedonian Academy of Sciences and Arts (see Figures 4 and 5). About 10 papers related to structural studies of Minerals from the Republic of Macedonia are published thereby, 7 of them being in collaboration with Croatian Crystallographers (e.g. Jovanovski *et al.*, 2005; Naumov *et al.*, 2007d; Jovanovski *et al.*, 2009a; Jovanovski *et al.*, 2009b; Makreski et al., 2009; Naumov *et al.*, 2010b).

Figure 4 Mineral kyanite, Al_2SiO_5 (Čumovo locality, Macedonia)

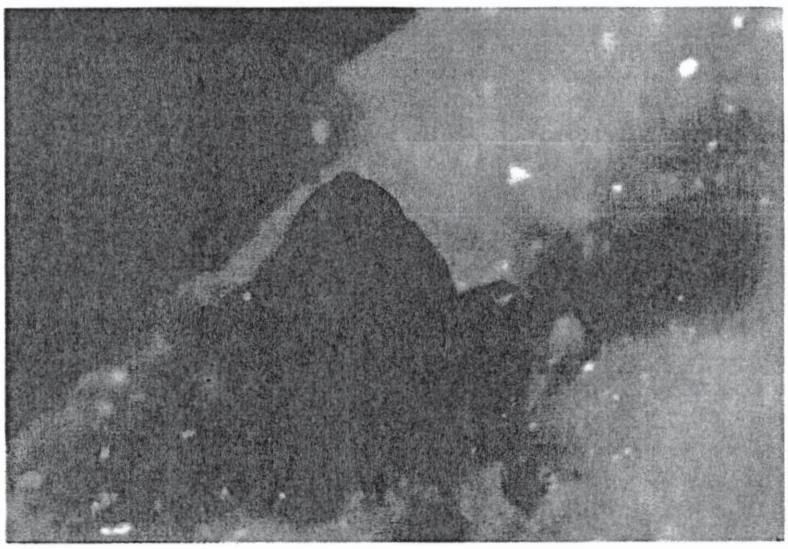

Figure 5 Mineral fluorite, CaF_2 (Sivec locality, Macedonia)

One of the most active research groups of the Division of Crystallography in Skopje (G. Jovanovski, O. Grupče, P. Naumov) has contributed to the crystallographic research with over 40 publications in renowned international journals, which are mainly related to the rich coordination chemistry of saccharin (artificial sweetener with suspected carcinogenic activity), thiosaccharin and their mixed complexes with aromatic bases (e.g. Jovanovski & Šoptrajanov, 1986; Jovanovski *et al.*, 1988a; Jovanovski *et al.*, 1988b; Hergold-Brundić *et al.*, 1989; Penavić *et al.*, 1990; Jovanovski *et al.*, 1990; Grupče *et al.*, 1994; Jovanovski *et al.*, 1998a; Jovanovski *et al.*, 1998b; Grupče *et al.*, 1999; Jovanovski *et al.*, 1999; Jovanovski, 2000; Naumov & Jovanovski, 2000; Naumov & Jovanovski, 2001; Naumov *et al.*, 2001;Naumov *et al.*, 2002c; Icbudak *et al.*, 2003; Jovanovski *et al.*, 2004; Makreski *et al.*, 2004; Naumov *et al.*, 2005c; Naumov *et al.*, 2006c). About 20 of the above mentioned papers are published in collaboration with Croatian crystallographers (D. Grdenić, B. Kamenar, M. Sikirica, B. Kaitner, M. Penavić, A. Hergold-Brundić, A. Nagl, D. Matković-Čalogović, V. Bermanec).

In 2000 P. Naumov moved to Tokyo Institute of Technology, where under the supervision of Yuji Ohashi, he received (2004) a PhD degree in diffraction methods. After forming a new laboratory for study of photoinduced phenomena and solid-state chemistry at the National Institute for Materials Science in Japan (2004—2007), since 2007 he is a leader of an independent research group at Osaka University (Japan). In 2009 Dr. Naumov was appointed Associate Professor from the external staff at the Institute of Chemistry in Skopje, continuing to collaborate with the researchers in Macedonia in the efforts to contribute to improvement of the conditions for crystallography research in the country. The current research interests of the group are related to materials exhibiting photoinduced and thermal phase transitions – promising candidates as future materials for the (opto)spintronics, X-ray photodiffraction and time-resolved diffraction using synchrotron X-rays (e.g. Wojciechowski *et al.*, 2001; Ng *et al.*, 2001; Naumov *et al.*, 2002b; Naumov & Ohashi, 2004a; Naumov & Ohashi, 2004b; Naumov et al., 2005a; Naumov *et al.*, 2005b; Naumov *et al.*, 2006a; Naumov *et al.*, 2006b; Naumov *et al.*, 2007a; Naumov *et al.*, 2007b; Naumov *et al.*, 2007c; Naumov & Belik, 2008; Naumov *et al.*, 2008; Naumov *et al.*, 2009a; Naumov *et al.*, 2009b; Yang *et al.*, 2009; Naumov *et al.*, 2010c; Naumov *et al.*, 2010d; Laguna *et al.*, 2010; Skoko *et al.*, 2010).

As one of the outstanding reported structures from the Macedonian crystallographers [P. Naumov (Skopje, Tokio), G. Jovanovski (Skopje), O. Grupče (Skopje), together with B. Kaitner (Zagreb), D. A. Rae (Canberra) and S. W. Ng (Kuala Lumpur)], is determined crystal structure of the sweetener sodium saccharinate, $Na_{64}(C_7H_4NO_3S)_{64}\cdot 120H_2O$ (Naumov et al. 2005), an important commercial compound for which a long time the formula of a dihydrate had been assumed (see Fig. 6). The extremely complicated modulated structure with a unit cell of 15.6 nm^3 and $Z' = 16$ ($Z = 64$) was ranked #6 in the World's database of small-molecule structures with high Z' (University of Durham).

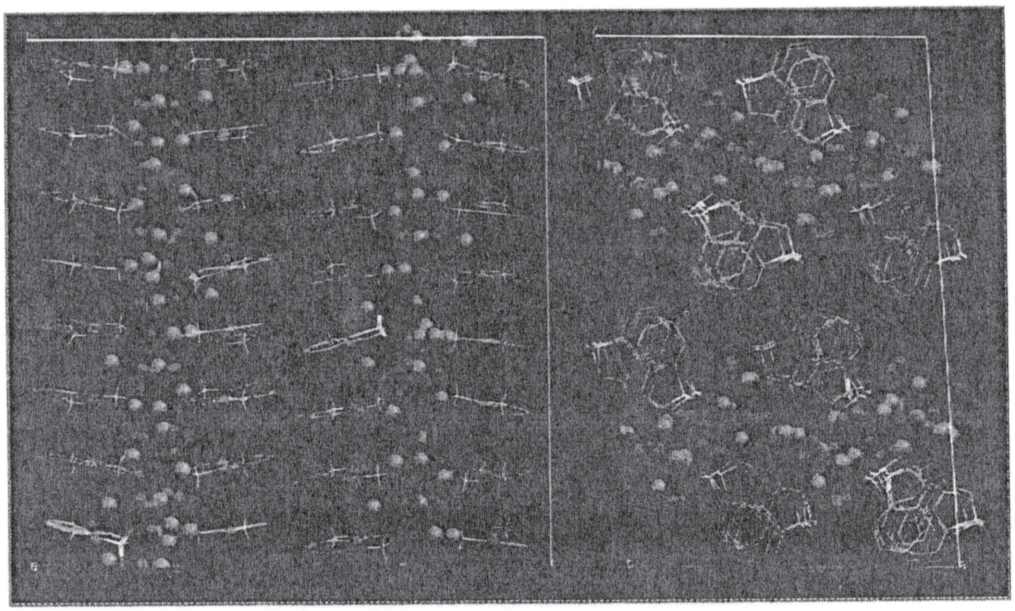

Figure 6 Crystal structure of the artificial sweetener sodium saccharinate, $Na_{64}(C_7H_4NO_3S)_{64}\cdot 120H_2O$ (Na-violet; O-red)

Another very recent example of the activity of the Division of Crystallography is the first direct observation of an all-solid autocatalytic reaction set, a group of photo-triggered reactions proceeding in the crystals of the natural mineral realgar (Naumov *et al.*, 2010) (see Fig. 7).

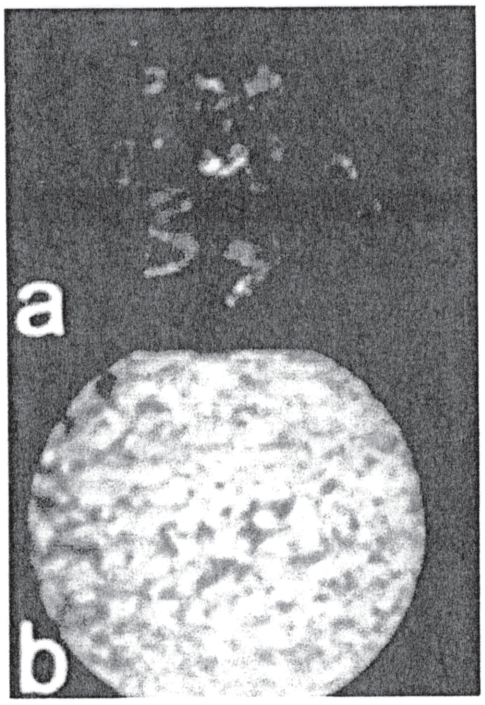

Figure 7 Crystals of Macedonian mineral realgar (As_4S_4) before exposure to visible light (a) and after photochemical conversion to pararealgar (b).

In summary, Macedonian crystallographers have published about 130 crystallographic papers, 36 of them (28%) being in collaboration with the Croatian crystallographers.

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