# ORIGINAL PAPER

# Synthesis and Crystal Structures of Methyl 3-(Benzoylamino)-6-methyl-2-oxo-2*H*-pyran-5-carboxylate and *N*-[5-(3,4-Dimethoxyphenyl)-6-methyl-2-oxo-2*H*-pyran-3-yl]benzamide

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Abstract The compounds methyl 3-(benzoylamino)-6methyl-2-oxo-2H-pyran-5-carboxylate (1), C<sub>15</sub>H<sub>13</sub>NO<sub>5</sub>, and *N*-[5-(3,4-dimethoxyphenyl)-6-methyl-2-oxo-2*H*-pyran-3-yl] benzamide (2),  $C_{21}H_{19}NO_5$ , crystallize as a centrosymmetric hydrogen-bonded dimer facilitated by N-H...O interactions involving the amide and carbonyl moiety of the lactone group of adjacent molecules. Supramolecular aggregation in **1** is controlled by a combination of  $\pi$ - $\pi$  interactions [centroid–centroid distance = 4.0745(11) Å] and weak C–H…O hydrogen bonding between the phenyl ring of the benzoylamino group and the carbonyl atom of the methoxycarbonyl group and in 2 by a combination of  $\pi$ - $\pi$  interactions [centroidcentroid distance = 4.0699(8) and 4.1556(10) Å], weak C-H...O interactions between the methoxy substituents of the adjacent dimethoxyphenyl group and weak C-H···  $\pi$ interactions.

**Keywords** 2*H*-Pyran-2-ones  $\cdot$  Crystal structure  $\cdot$ Hydrogen bonds  $\cdot$  C–H $\cdots \pi$  interaction  $\cdot \pi - \pi$  interaction

# Introduction

2*H*-Pyran-2-ones represent an important class of compounds useful for various ring transformations [1, 2], especially for those including nucleophiles [3] and as dienes in Diels–Alder reactions [4]. Considering the recent

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When for cycloadditions with 2H-pyran-2-ones alkynes are used as dienophiles the initial step gives the 7-oxabicyclo[2.2.2]octadiene adducts, which are, however, unstable and spontaneously eliminate carbon dioxide to yield the final benzene derivatives [10]. On the other hand, when alkenes are applied as dienophiles, the initially formed 7-oxabicyclo[2.2.2]octenes are more stable and can be in some cases isolated [11, 12]. Nevertheless, under thermal reaction conditions the carbon dioxide is also often eliminated yielding cyclohexadiene systems, which can either serve as another diene to produce bicyclo[2.2.2]octene products (double cycloadducts) [13] or can be oxidized (aromatized) into benzene derivatives (formally analogous to those obtained with alkynes). These oxidations can be facilitated by the application of a heterogeneous dehydrogenation catalyst (such as Rh/C) [14, 15]. Bicyclo[2.2.2]octenes easily prepared from 2H-pyran-2-ones according to this methodology represent a very interesting set of compounds for further studies of desymmetrization [16] and also as rewarding systems for the investigations of steric effects exhibited by fused rings of different sizes on the stereochemistry of cycloadditions of maleic anhydride and N-substituted maleimides [17]. Additionally, 2Hpyran-2-ones can be used as useful precursors for the preparation of substituted indoles [18].

Due to the wide applicability of 2*H*-pyran-2-ones, a straightforward one-pot synthesis starting from the simple commercially available compounds was desired [19–21].

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Examples given here include such a transformation between a molecule containing an activated  $CH_2$  group, a  $C_1$ -synthon *N*,*N*-dimethylformamide dimethyl acetal (DMFDMA) and hippuric acid as an *N*-acylglycine derivative taking place in acetic anhydride (or in a mixture with acetic acid) as the solvent yielding methyl 3-(benzoylamino)-6-methyl-2-oxo-2*H*-pyran-5-carboxylate (1) and *N*-[5-(3,4-dimethoxyphenyl)-6-methyl-2-oxo-2*H*-pyran-3-yl] benzamide (2) (Scheme 1).

## Experimental

#### Materials and Measurements

Melting points were determined on a micro hot stage apparatus and are uncorrected. <sup>1</sup>H NMR spectra were recorded with a Bruker Avance DPX 300 spectrometer at 29 °C and 300 MHz using Me<sub>4</sub>Si as an internal standard. <sup>13</sup>C NMR spectra were recorded on the same instrument at 75.5 MHz and are referenced against the central line of the solvent signal (CDCl<sub>3</sub> triplet at 77.0 ppm). IR spectra were obtained with a Bio-Rad FTS 3000MX as KBr pellets. MS spectra were recorded with a VG-Analytical AutoSpec Q instrument. Elemental analyses (C, H, N) were performed with a Perkin Elmer 2400 Series II CHNS/O Analyzer. TLC was carried out on Fluka silica-gel TLC-cards. All reagents and solvents were used as received from commercial suppliers.

Synthesis of Methyl 3-(benzoylamino)-6-methyl-2-oxo-2*H*-pyran-5-carboxylate (1)

A mixture of methyl acetoacetate (23.22 g, 0.2 mol), DMFDMA (23.83 g, 0.2 mol), acetic anhydride (200 mL) and glacial acetic acid (50 mL) was heated on an oil bath at 80 °C for 150 min. Thereafter, hippuric acid (35.84 g, 0.2 mol) was added and heated on an oil bath at 80 °C for further 4 h. After the removal of the volatile components the residue was treated with ethanol (125 mL) and, after cooling, the precipitate was filtered off and washed with a small amount of ethanol (10 mL) to afford 35.93 g (63%) of the title product **1**. M.p. 164–167.5 °C (EtOH). Anal. calcd. for  $C_{15}H_{13}NO_5$ : C, 62.72; H, 4.56; N, 4.88. Found: C, 63.00; H,



Scheme 1 Molecular structures of 1 and 2

4.49; N, 4.93. IR (KBr) 3370, 1735, 1711, 1674, 1638, 1600, 1580, 1531, 1492 cm<sup>-1</sup>. <sup>1</sup>H NMR (300 MHz, CDCl<sub>3</sub>)  $\delta$  2.69 (s, 3H, Me), 3.90 (s, 3H, CO<sub>2</sub>Me), 7.54 (m, 3H, Ph), 7.88 (m, 2H, Ph), 8.54 (s, 1H, NH), 8.87 (s, 1H, 4-H). <sup>13</sup>C NMR (75.5 MHz, CDCl<sub>3</sub>)  $\delta$  19.3, 52.2, 109.5, 122.1, 123.6, 126.9, 128.7, 132.3, 133.2, 158.5, 162.8, 164.1, 165.6.

Synthesis of *N*-[5-(3,4-Dimethoxyphenyl)-6-methyl-2oxo-2*H*-pyran-3-yl]benzamide (**2**)

A mixture of (3,4-dimethoxyphenyl)acetone (16.68 g, 0.086 mol) and DMFDMA (21.46 g, 0.18 mol) was heated on reflux for 4 h. The volatile components were removed under reduced pressure, thereafter hippuric acid (15.81 g, 0.088 mol) and acetic anhydride (111 mL) were added and the mixture heated on an oil bath at 90 °C for further 4 h. After the removal of the volatile components the residue was treated with ethanol (55 mL) and, after cooling, the precipitate was filtered off and washed with a small amount of ethanol (5 mL) to afford 18.384 g (59%) of the title product **2**. M.p. 161–162 °C (EtOH). [21].

## Crystallography

Single-crystal X-ray diffraction data were collected at room temperature on a Nonius Kappa CCD diffractometer with the graphite monochromated Mo-K $\alpha$  radiation  $(\lambda = 0.71073 \text{ Å})$ . The data were processed by DENZO [22]. Structures were solved by direct methods implemented in SHELXS-97 and refined by a full-matrix leastsquares procedure based on  $F^2$  with SHELXL-97 [23]. All non-hydrogen atoms were refined anisotropically. All hydrogen atoms were readily located in a difference Fourier maps and were subsequently treated as riding atoms in geometrically idealized positions, with C-H = 0.93 (aromatic and alkenyl) or 0.96 Å (CH<sub>3</sub>), N–H = 0.86 Å and with  $U_{iso}(H) = kU_{eq}(C \text{ or } N)$ , where k = 1.5 for NH and methyl groups, which were permitted to rotate but not to tilt, and 1.2 for all other H atoms. Crystallographic data are listed in Table 1. Selected bond lengths and bond angles are listed in Table 2. All the bond lengths of compounds 1 and 2 are within normal ranges [24]. Hydrogen bonding interactions are listed in Table 3.

## **Results and Discussion**

In both compounds **1** and **2** the central 2*H*-pyran-2-one ring is planar (Fig. 1). The maximum deviation from the mean plane described by the ring atoms is 0.040(2) Å for atom C1 in **1** and -0.017(1) Å in **2**, again for atom C1. In both crystal structures weak intramolecular C3–H3…O3

Table 1Crystal data and refinement parameters for the compounds 1and 2

| Compound                                     | 1   | 2                                  |
|--|---|------------------------------------|
| CCDC   | 834643  | 834644                             |
| Molecular formula                            | C <sub>15</sub> H <sub>13</sub> NO <sub>5</sub> | C21H19NO5                          |
| Molecular weight                             | 287.26  | 365.37                             |
| Crystal system                               | Triclinic                                       | Triclinic                          |
| Space group                                  | P - 1   | P - 1                              |
| Temperature (K)                              | 293(2)  | 293(2)                             |
| a (Å)  | 8.4667(3)                                       | 8.1480(2)                          |
| b (Å)  | 8.4753(3)                                       | 9.5704(3)                          |
| c (Å)  | 10.9239(4)                                      | 12.2802(4)                         |
| α (°)  | 86.481(2)                                       | 83.255(2)                          |
| β (°)  | 72.282(2)                                       | 73.079(2)                          |
| γ (°)  | 64.194(2)                                       | 77.758(2)                          |
| $V(\text{\AA}^3)$                            | 669.91(4)                                       | 893.72(5)                          |
| Ζ  | 2   | 2                                  |
| $D_{\text{calc}} (\text{g cm}^{-3})$         | 1.424   | 1.358                              |
| Absorption coefficient (mm <sup>-1</sup> )   | 0.108   | 0.097                              |
| <i>F</i> (000)                               | 300   | 384                                |
| Crystal dimensions (mm)                      | $0.50 \times 0.25 \times 0.25$                  | $0.40 \times 0.20 \times 0.13$     |
| Theta range (°)                              | 5.47-27.47                                      | 3.55-27.46                         |
| Range/indices (h, k, l)                      | -10, 10; -10,<br>10; -14, 14                    | -10, 10; -12,<br>12; -15, 15       |
| Reflections collected                        | 5390  | 6954                               |
| Independent reflections                      | 3008<br>[ $R_{\rm int} = 0.0193$ ]              | 4030<br>[ $R_{\rm int} = 0.0158$ ] |
| Completeness                                 | 98.2%   | 98.4%                              |
| $T_{\rm max}/T_{\rm min}$                    | 0.9734/0.9478                                   | 0.9874/0.9621                      |
| Data/restraints/parameters                   | 3008/0/193                                      | 4030/0/248                         |
| $R_1, wR_2 [I > 2\sigma(I)]^a$               | 0.0446, 0.1187                                  | 0.0464, 0.1193                     |
| $R_1, wR_2$ (all data) <sup>b</sup>          | 0.0551, 0.1297                                  | 0.0608, 0.1337                     |
| Goodness of fit on $F^2$ , $S^c$             | 1.037   | 1.017                              |
| Largest diff. peak<br>and hole (e $Å^{-3}$ ) | 0.222 and -0.213                                | 0.156  and  -0.188                 |

 ${}^{a}R = \sum ||F_{o}| - |F_{c}|| \sum F_{o}|, \ ^{b}wR_{2} = \{\sum [w(F_{o}^{2} - F_{c}^{2})^{2}] / \sum [w(F_{o}^{2})^{2}] \}^{1/2}, \ ^{c}S = \{\sum [(F_{o}^{2} - F_{c}^{2})^{2}] / (n/p) \}^{1/2} \text{ where } n \text{ is the number of reflections and } p \text{ is the total number of parameters refined}$ 

hydrogen bonding is present. Furthermore, in compound 1 additional C6–H6C···O4 hydrogen bonding is present (Table 3).

The mean plane through the amide group (O3-C7-N1-C2-C8) in **1** is inclined to the 2*H*-pyran-2-one ring by 20.38(8)° and the dihedral angle between the 2*H*-pyran-2-one and phenyl ring (C8-C13) is 33.35(9)° while these angles in **2** are 12.15(8)° and 23.54(8)°, respectively. This deviation of the amide group from planarity is similar also in 3-benzoylamino-7,7-dimethyl-5-oxo-5,6,7,8-tetrahydro-coumarin being 12.95° [25], while in the other two known

445

|         |          |      |         | 0   |            |     |       |         |
|---------|----------|------|---------|-----|------------|-----|-------|---------|
| Table 2 | Selected | bond | lengths | [A] | and angles | [°] | for 1 | and $2$ |

| 1      |            |          |            |
|--------|------------|----------|------------|
| 01–C1  | 1.3786(15) | C501C1   | 124.25(10) |
| O1-C5  | 1.3653(16) | O2-C1-O1 | 117.48(11) |
| O2–C1  | 1.2055(16) | O2-C1-C2 | 126.53(12) |
| O3–C7  | 1.2173(17) | C7-N1-C2 | 125.25(11) |
| O4-C14 | 1.2019(17) | C3-C2-N1 | 127.05(12) |
| O5-C14 | 1.3316(17) | N1-C2-C1 | 113.14(11) |
| O5-C15 | 1.4476(17) | O3-C7-N1 | 122.00(12) |
| N1-C7  | 1.3661(17) | O3–C7–C8 | 121.66(12) |
| N1-C2  | 1.4017(16) |          |            |
| 2      |            |          |            |
| 01–C1  | 1.3645(16) | C1O1C5   | 123.45(11) |
| O1-C5  | 1.3800(16) | O2-C1-O1 | 118.32(12) |
| O2-C1  | 1.2091(17) | O2-C1-C2 | 125.14(12) |
| O3–C7  | 1.2206(18) | C7-N1-C2 | 126.00(12) |
| O4-C16 | 1.3636(16) | C3-C2-N1 | 127.63(13) |
| O5-C17 | 1.3605(16) | N1-C2-C1 | 112.59(12) |
| N1-C7  | 1.3616(18) | O3-C7-N1 | 121.39(13) |
| N1-C2  | 1.4015(17) | O3–C7–C8 | 121.71(13) |
|        |            |          |            |

structures it is much smaller,  $4.17^{\circ}$  [26] and  $4.95^{\circ}$  [27]. The dihedral angle between the 2*H*-pyran-2-one and phenyl ring of the benzoylamino group are in all three known structures markedly smaller, ranging from  $6.09^{\circ}$  to  $7.80^{\circ}$  [25–27].

The methoxycarbonyl group (C15/O5/O4/C14/C4) in **1** is nearly planar with the central 2*H*-pyran-2-one ring, the dihedral angle is 9.09(8)°. Such small deviations of carboxylic group (either as an acid or ester functionality) from the planarity of 2*H*-pyran-2-one [28–34] or benzene ring [35–43] are often observed. When a bulky substituent is present in the *ortho* position to the carboxylic group much larger twist angles ranging from ~20° up to ~80° are observed in the cases of 2*H*-pyran-2-one [44–48] or benzene ring [49–58].

In the compound **2** a dimethoxyphenyl group is attached to the central 2*H*-pyran-2-one ring instead of methoxycarbonyl group. The dihedral angle between the 2*H*-pyran-2-one and phenyl ring (C14–C19) is  $55.99(7)^{\circ}$ . In CSD we found no structure similar to **2** that would incorporate a dimethoxyphenyl group at the position 5 of pyran-2-one ring (as in **2**) and neither at the positions 3 or 4. However, a few structures that possess a dimethoxyphenyl group attached at the position 6 of pyran-2-one ring were found [59–62]. Wide variety of dihedral angles can be observed in these cases, being either small (4.56° [59], 8.83° [60]) or large (19.58° [61], 42.54° [62]).

Both compounds 1 and 2 crystallize as centrosymmetric hydrogen-bonded dimers facilitated by the amide and carbonyl moiety of the lactone group of adjacent molecules

Table 3Hydrogen bondgeometry of 1 and 2 (Å and °)

| D–H…A         | D–H (Å) | H…A (Å) | D…A (Å)    | D–H···A (°) | Symmetry code       |
|---------------|---------|---------|------------|-------------|---------------------|
| 1             |         |         |            |             |                     |
| N1-H1O2       | 0.86    | 2.42    | 3.2517(15) | 162.8       | 1 - x, 1 - y, 1 - z |
| С3-Н3…О3      | 0.93    | 2.31    | 2.843(2)   | 115.7       |                     |
| С6-Н6С…О4     | 0.96    | 2.20    | 2.890(2)   | 128.0       |                     |
| C11-H11O4     | 0.93    | 2.54    | 3.4321(19) | 161.1       | x, 1 + y, 1 + z     |
| C13-H13····O2 | 0.93    | 2.43    | 3.3458(18) | 167.6       | 1 - x, 1 - y, 1 - z |
| 2             |         |         |            |             |                     |
| N1-H1O2       | 0.86    | 2.52    | 3.3303(17) | 156.5       | -x, 1 - y, 2 - z    |
| С3-Н3…О3      | 0.93    | 2.26    | 2.8286(18) | 118.8       |                     |
| C13-H13····O2 | 0.93    | 2.38    | 3.312(2)   | 176.5       | -x, 1 - y, 2 - z    |
| C21-H21BO4    | 0.96    | 2.54    | 3.423(2)   | 153.1       | -x, -1 - y, 1 - z   |
| C20–H20C…Cg3  | 0.96    | 2.94    | 3.738(2)   | 141.0       | -x, -y, 1 - z       |
|               |         |         |            |             |                     |



Fig. 1 The molecular structure of 1 and 2, showing the atomlabelling scheme. Displacement ellipsoids are drawn at the 50% probability level

(Figs. 2,3; Table 3). The dimers in 1 and 2 can be described by the graph-set motif  $R_2^2(10)$  [63]. Dimers in both crystal structures are further stabilized by weak C13–H13…O2 interactions. In both compounds the two 2*H*-pyran-2-one rings involved in the dimer formation are not coplanar. The separation of mean planes, which are described by the atoms of each 2*H*-pyran-2-one ring in a dimer is 1.431 Å in 1 and 1.569 Å in 2. In the Cambridge Structural Database (CSD) [64] we found only three structures with benzoylamino moiety attached to the 2*H*-pyran-2-one ring, in two of them this moiety is attached at

J Chem Crystallogr (2012) 42:443-449

the position 3 [25, 26] and in one structure at the position 6 [27]. Similar dimer formation is present only in 3-benzoylamino-7,7-dimethyl-5-oxo-5,6,7,8-tetrahydrocoumarin with the separation of mean planes through the 2*H*-pyran-2-one ring of adjacent molecules of 1.595 Å [25]. In other two compounds dimer formation is not present because N–H group is either not involved in the hydrogen bonding [26] or is hydrogen-bonded to a 1,4-dioxane solvate molecule [27].

Dimers of **1** form ribbons by weak C11–H11····O4 (x, 1 + y, 1 + z) hydrogen bonding between the phenyl ring of the benzoylamino group and the carbonyl atom of the methoxycarbonyl group parallel to [011] (Fig. 2; Table 3). Supramolecular aggregation is stabilized by  $\pi$ – $\pi$  interactions between two parallel C8–C13 rings (centroid Cg1), with a Cg1···Cg1(1 - x, 2 - y, 1 - z) centroid-to-centroid distance of 4.0745(11) Å, a perpendicular distance from the centroid Cg1 to the plane of the other ring of 3.7354(8) Å and a centroid offset of 1.627 Å (Fig. 4).

Dimers of 2 also form ribbons by weak C21-H21B····O4 (-x, -1 - y, 1 - z) hydrogen bonding, but in this case between the methoxy substituents of the adjacent dimethoxyphenyl group parallel to [011] (Fig. 3; Table 3). Supramolecular aggregation is stabilized by weak C20-H20C···Cg3(-x, -y, 1 - z) interactions (Cg3 is the centroid of the C14–C19 ring) (Fig. 5) and  $\pi$ – $\pi$  interactions parallel to [100] between two parallel O1/C1-C5 rings (centroid Cg1), with a Cg1...Cg1(-x, -y, 2 - z) centroidto-centroid distance of 4.0699(8) Å, a perpendicular distance from the centroid Cg1 to the plane of the other ring of 3.5648(6) Å and a centroid offset of 1.964 Å. Supramolecular aggregation is further stabilized by  $\pi - \pi$  interactions between O1/C1–C5 ring (centroid Cg1) and C8–C13 ring (centroid Cg2), with a  $Cg1\cdots Cg2(-1 + x, y, z)$  centroid-tocentroid distance of 4.1556(10) Å, a dihedral angle between the rings of  $23.54(8)^\circ$ , a perpendicular distance from the centroid Cg1 to the plane of the other ring of 2.8300(6) Å



Fig. 3 1D framework formation of dimers in 2. Dashed lines indicate hydrogen bonds, dimers are connected via C21-H21B···O4 hydrogenbonding



Fig. 4 A packing diagram for 1. Dashed lines indicate  $\pi$ - $\pi$  interactions and C11-H11...O4 hydrogen bonds. For the sake of clarity, H atoms not involved in the motif shown have been omitted



**Fig. 5** A packing diagram for **2**. *Dashed lines* indicate C20– $H20C\cdots Cg3$  hydrogen-bonding

and the angle between the intercentroid vector and the normal to the second ring of 23.54° (consequently the perpendicular distance from the centroid *Cg*2 to the plane of the first ring is 3.8096(7) Å and the angle between the intercentroid vector and the normal to the first ring is 47.08°) (Fig. 6). These  $\pi$ - $\pi$  interactions are consistent with well defined  $\pi$ - $\pi$  stacking interactions [65–68]. According to Janiak [69], they can be regarded as medium strong, since strong interactions exhibit rather short centroid–centroid contacts (*Cg*...*Cg* < 3.8 Å), small slip angles (<25°) and small vertical displacements (<1.5 Å), which translate into a sizeable overlap of the aromatic planes. In comparison, medium-to-weak interactions exhibit rather long centroid– centroid distances (>4.0 Å) together with large slip angles (>30°) and large vertical displacements (>2.0 Å) [69–71]. **Fig. 6** A packing diagram for **2**. *Dashed lines* indicate  $\pi - \pi$  interactions. For the sake of clarity, H atoms have been omitted



#### **Supplementary Material**

Crystallographic data of **1** and **2** were deposited in the Cambridge Crystallographic Data Center under the number CCDC 834643-834644. CIF files containing complete information on the studied structures may be obtained free of charge from the Director, CCDC, 12 Union Road, Cambridge, CB2 1EZ, UK, fax +44-1223-336033; e-mail: data\_request@ccdc.cam.ac.uk or from the following web site: www.ccdc.cam.ac.uk/data\_request/cif.

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## References

- Afarinkia K, Vinader V, Nelson TD, Posner GH (1992) Tetrahedron 48:9111–9171
- 2. Goel A, Ram VJ (2009) Tetrahedron 65:7865–7913
- 3. Požgan F, Kočevar M (2009) Heterocycles 77:657-678
- 4. Kranjc K, Kočevar M (2010) Curr Org Chem 14:1050-1074
- 5. Anastas P, Warner J (1998) Green chemistry: theory and practice. Oxford University Press, New York
- 6. Polshettiwar V, Varma RS (2008) Chem Soc Rev 37:1546-1557
- 7. Gupta M, Paul S, Gupta R (2009) Acta Chim Slov 56:749-764
- 8. Li C-J, Chen L (2006) Chem Soc Rev 35:68-82
- Martins MAP, Frizzo CP, Moreira DN, Buriol L, Machado P (2009) Chem Rev 109:4140–4182
- 10. Kranjc K, Kočevar M (2005) New J Chem 29:1027-1034
- Augustyns B, Maulide N, Markó IE (2005) Tetrahedron Lett 46:3895–3899
- Juranovič A, Kranjc K, Perdih F, Polanc S, Kočevar M (2011) Tetrahedron 67:3490–3500
- 13. Kranjc K, Kočevar M (2007) Bull Chem Soc Jpn 80:2001-2007
- Hoheisel TN, Schrettl S, Szilluweit R, Frauenrath H (2010) Angew Chem Int Ed 49:6496–6515
- 15. Kranjc K, Polanc S, Kočevar M (2003) Org Lett 5:2833-2836

- Hren J, Perdih F, Polanc S, Kočevar M (2011) Eur J Org Chem 3368–3374
- 17. Kranjc K, Perdih F, Kočevar M (2009) J Org Chem 74: 6303–6306
- 18. Kranjc K, Kočevar M (2008) Tetrahedron 64:45-52
- Kepe V, Kočevar M, Polanc S, Verček B, Tišler M (1990) Tetrahedron 46:2081–2088
- Kočevar M, Polanc S, Verček B, Tišler M (1990) Liebigs Ann Chem 501–503
- 21. Požgan F, Kranjc K, Kepe V, Polanc S, Kočevar M (2007) Arkivoc (viii), 97-111
- Otwinowski Z, Minor W (1997) Methods in enzymology. In: Carter Jr CW, Sweet RM (eds) Macromolecular crystallography, part A, vol 276, pp 307–326. Academic Press, New York
- 23. Sheldrick GM (2008) Acta Cryst A64:112-122
- 24. Allen FH, Kennard O, Watson DG, Brammer L, Orpen AG, Taylor R (1987) J Chem Soc Perkin Trans II:S1–S19
- 25. Leban I, Kočevar M, Verček B (1993) Acta Cryst C49:343-345
- Štefane B, Perdih A, Pevec A, Šolmajer T, Kočevar M (2010) Eur J Org Chem 5870–5883
- Zakhs VE, Yakovlev IP, Gindin VA, Chumakov YM, Biyushkin VN, Malinovskii TI, Ivin BA (1992) Zh Org Khim 28:2611–2619
- 28. Song JH, Lei YX, Rappoport Z (2007) J Org Chem 72: 9152–9162
- 29. Hossain MB, van der Helm D (1987) Acta Cryst C43:992-994
- Michinobu T, Bito M, Yamada Y, Katayama Y, Noguchi K, Masai E, Nakamura M, Ohara S, Shigehara K (2007) Bull Chem Soc Jpn 80:2436–2442
- Brbot-Šaranović A, Pavlović G, Cindrić M (2000) Struct Chem 11:65–76
- Cindrić M, Vrdoljak V, Kajfež T, Novak P, Brbot-Šaranović A, Strukan N, Kamenar B (2002) Inorg Chim Acta 328:23–32
- Brbot-Šaranović A, Pavlović G, Vrdoljak V, Cindrić M (2001) Croat Chem Acta 74:441–454
- Wiedemann D, Grohmann A (2009) Z Naturforsch B Chem Sci 64:1276–1288
- 35. Prior TJ, Sharp AJ (2010) J Chem Crystallogr 40:630-633
- Hussain M, Ali S, Karmazin Brelot v, Stoeckli-Evans H (2006) Acta Cryst E62:2657–2659
- 37. Xia M, Ma K-R, Zhu Y (2010) J Chem Crystallogr 40:634-638
- 38. Batsanov AS (2004) Acta Cryst E60:1948–1949
- 39. Obreza A, Perdih F (2012) J Struc Chem 53 (accepted for publication)
- Zhong G-X, Hu H-D, Xia C-N, Jiang J-S, Chen T–T (2010) J Chem Crystallogr 40:735–739
- Cai J, Zhou W, Chen JQ, Sun M, Ji M (2009) J Chem Crystallogr 39:108–111

- Peori MB, Vaughan K, Bertolasi V (2009) J Chem Crystallogr 39:178–181
- 43. Kranjc K, Kočevar M, Perdih F (2011) Acta Cryst C67:o201o205
- 44. Pfeffer M, Rotteveel MA, de Cian A, Fisher J, le Borgne G (1991) J Organomet Chem 413:C15–C19
- Covarrubias-Zúñiga A, Espinosa-Pérez G (1997) Anal Sci 13: 683–684
- 46. Baker SR, Begley MJ, Crombie L (1981) J Chem Soc Perkin Trans 1:182–189
- Anary-Abbasinejad M, Anaraki-Ardakani H, Mosslemin MH, Khavasi HR (2010) J Braz Chem Soc 21:319–323
- Calvert JL, Hartshorn MP, Robinson WT, Wright GJ (1992) Aust J Chem 45:361–370
- Boyarskiy VP, Fonari MS, Suwinska K, Simonov YA (2009) J Struct Chem 50:585–587
- Rybalova TV, Gatilov YV, Zonov YV, Karpov VM (2008) J Struct Chem 49:742–747
- 51. Lalancette RA, Vanderhoff PA, Thompson HW (1990) Acta Cryst C46:1682–1686
- Gossage RA, Jenkins HA, Quail JW (2010) J Chem Crystallogr 40:272–277
- Smith G, Wermuth UD, White JM (2009) Acta Cryst C65:o103– o107
- 54. Ru Z-L, Wang G-X (2009) Acta Cryst E65:o3264
- 55. Raza AR, Saddiqa A, Tahir MN, Danish M, Iqbal MS (2010) Acta Cryst E66:0288
- Medimagh R, Marque S, Prim D, Marrot J, Chatti S (2009) Org Lett 11:1817–1820

- 57. Zou P, Luo S-N, Xie M-H, Liu Y-L, Wu J (2010) Acta Cryst E66:0360
- 58. Zhu X-Y, Gao G-W, Men J, Ng SW (2009) Acta Cryst E65:o794
- Robinson PD, Beatty A, Hua DH, Chen Y, Meyers CY, Perchellet EM, Ladesich JB, Perchellet J-P (1999) Acta Cryst C55: 1188–1190
- Hsung RP, Cole KP, Zehnder LR, Wang J, Wei L–L, Yang X-F, Coverdale HA (2003) Tetrahedron 59:311–324
- Shen HC, Wang J, Cole KP, McLaughlin MJ, Morgan CD, Douglas CJ, Hsung RP, Coverdale HA, Gerasyuto AI, Hahn JM, Liu J, Sklenicka HM, Wei L–L, Zehnder LR, Zificsak CA (2003) J Org Chem 68:1729–1735
- Williams CM, Heim R, Bernhardt PV (2005) Tetrahedron 61: 3771–3779
- Bernstein J, Davis RE, Shimoni L, Chang N-L (1995) Angew Chem Int Ed Engl 34:1555–1573
- Cambridge Structural Database (2002) CSD, v5.32 (May 2011 update) see Allen FH (2002) Acta Cryst B58:380–388
- 65. Hunter CA, Sanders JKM (1990) J Am Chem Soc 112: 5525–5534
- 66. Hunter CA (1994) Chem Soc Rev 23:101-109
- 67. Choudhury RR, Chitra R (2010) CrystEngComm 12:2113-2121
- 68. Perdih F, Perdih A (2011) Cellulose 18:1139–1150
- 69. Janiak C (2000) J Chem Soc Dalton Trans 3885-3896
- 70. Dorn T, Janiak C, Shandi A-K (2005) CrystEngComm 7:633-641
- Yang X-J, Drepper F, Wu B, Sun W-H, Haehnel W, Janiak C (2005) Dalton Trans 256–267