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A Closer Look at the Hydroiodination of Propiolic Acid

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Abstract: The crystal structures of *cis*-3-iodoacrylic acid (1), *trans*-3-iodoacrylic acid (2), *trans*-3-iodoacrylic acid methyl ester (3), 3,3-diiodopropanoic acid (4), and *trans*-2,3-diiodoacrylic acid (5) are reported. Compounds 1 and 2 are the kinetic and thermodynamic products, respectively, of the hydroiodination of propiolic acid. Compound 4 results from addition of a second equivalent of hydroiodic acid to 1 or 2, whereas 5 results from the addition of trace elemental iodine to propiolic acid.

Keywords: cis-3-Iodoacrylic acid, hydroiodination, propiolic acid, trans-3-iodoacrylic acid

INTRODUCTION

Although the syntheses of *cis*- and *trans*-3-iodoacrylic acid via the hydroiodination of propiolic acid were first described more than 120 years ago,^[1] it has not been until the more recent work by Moss et al.,^[2] Abarbri et al.,^[3] and Zoller and Uguen^[4] that procedures optimized for the isolation of one or the other stereoisomer have been available. The *cis* isomer, which is the kinetic product of hydroiodination, is preferentially formed when relatively mild reaction conditions are employed (8–10°C, CuI catalysis,^[2] or 4 h at $65^{\circ}C^{[3]}$), whereas formation of the thermodynamically favored *trans* isomer is promoted by more vigorous conditions (16 h at $130^{\circ}C^{[3]}$ or 57% HI solution starting with isolated *cis* isomer, 85–90°C, 24 h^[4]). Since

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these reports describing procedures for the synthesis of pure *cis*- or *trans*-3-iodoacrylic acid, these compounds have been increasingly employed as starting materials and building blocks in the synthesis of complex organic molecules with important pharmacological or medicinal properties. The value of these molecules as starting materials arises from the facility with which they undergo carbon–carbon bond forming reactions via the Stille coupling protocol.^[5] Examples of important natural products whose synthesis has been accomplished using *cis*- or *trans*-3-iodoacrylic acid, or their methyl esters, include strychnine,^[6,7] cylindramide,^[8] freelingyne (a sesquiterpene),^[9] xerulin (a cholesterol biosynthesis inhibitor),^[10,11] the macrolides elaiophylin^[12] and elaiolide^[13] (antibiotics and anthelmintics), (–)-macrolactin A (an antiviral agent),^[14,15] (+)-brefeldin C^[16] and (+)-brefeldin A^[17,18] (antiviral and antitumour agents), eunicenone A,^[19] (+)-aloperine (an anti-inflammatory agent),^[20] and formamicin (an antifungal, antibacterial, and antitumour agent).

In the course of earlier work with polyunsaturated dicarboxylates as connectors between dimolybdenum units,^[23] trans-3-iodoacrylic acid was prepared by the hydroiodination of propiolic acid. While following a literature procedure,^[4] it was noted that this reaction is attended by the formation of other products in greater or lesser amounts depending upon the specific conditions. Because of the importance of cis and trans iodoacrylic acids as organic synthons, and because they had not previously been characterized by crystallography, X-ray crystal structure determinations of these two simple organic acids were undertaken. Two other organic acids that were unexpected by-products of this reaction were identified and serve to emphasize the importance of precise stoichiometry and reaction temperature upon the product manifold. Reported here are the crystal structures of both cis- and trans-3-iodoacrylic acid, the methyl ester of the latter, and the structures of the unexpected by-products 3,3-diiodopropanoic acid and trans-2,3diiodoacrylic acid. The crystal structure of trans-3-iodoacrylic acid has been briefly described in an earlier publication.^[23]

RESULTS AND DISCUSSION

Figure 1 presents the structures of *cis*- and *trans*-3-iodoacrylic acid [(1) and (2), respectively], with crystal data and data collection and refinement details presented in Table 1. Bond lengths and angles are collected in Tables 2 and 3. The carbon-iodine bond lengths [2.070(4) and 2.077(6) Å] and the carbon–carbon double bond lengths [1.321(6) and 1.317(8) Å] are unexceptional. As is typically the case, these simple acids crystallize in a "head-to-head" fashion though hydrogen bonding in the carboxyl groups. The methyl ester of *trans*-3-iodoacrylic acid (3), prepared by refluxing the acid in MeOH with H₂SO₄ as catalyst (see Fig. 2), is a low melting solid that crystallizes upon slow cooling of the melt. Its crystallization was



Figure 1. Thermal ellipsoid plots of *cis*-3-iodoacrylic acid (1), *trans*-3-iodoacrylic acid (2), *trans*-3-iodoacrylic acid methyl ester (3), 3,3-diiodopropanoic acid (4) and *trans*-2,3-diiodoacrylic acid (5). Ellipsoids are drawn at the 50% probability level, except for 5, which has ellipsoids drawn at the 30% level.

an unexpected result because existing information in the literature on this compound appears to suggest that it is an oil at ambient temperature and pressure.^[24] Figure 1 illustrates a thermal ellipsoid plot of *trans*-3-iodoacrylic acid methyl ester (**3**), and Table 4 presents selected bond distances and angles.

If the hydroiodination of propiolic acid is attempted at temperatures greater than 100° C with excess HI, the *trans*-3-iodocrylic acid that is isolated is mixed with substantial quantities of another product from which it is readily distinguished by crystal morphology. The desired *trans*-3-iodoacrylic acid crystallizes as large, flat needle crystals, whereas the second product crystallizes as large, white, block-shaped crystals. The ¹H NMR spectrum of this second species identifies it as 3,3-diiodopropanoic acid (4), a product resulting from the addition of a second equivalent of HI

Parameter	cis-3-Iodoacrylic acid, 1	<i>trans</i> -3-Iodoacrylic acid, 2	<i>trans</i> -3-Iodoacrylic acid methyl ester, 3	3,3-Diiodo- propanoic acid, 4	<i>trans</i> -2,3-Diio- doacrylic acid, 5
Formula	C ₃ H ₃ IO ₂	C ₃ H ₃ IO ₂	C ₄ H ₅ IO ₂	$C_3H_4I_2O_2$	$C_3H_2I_2O_2$
FW	197.95	197.95	211.98	325.86	323.85
Crystal color	White	White	White	White	White
Crystal morphology	Plate	Needle	Prism	Block	Needle
Crystal system	Monoclinic	Orthorhombic	Orthorhombic	Monoclinic	Triclinic
Space group	$P2_1/n$	Cmca	$Cmc2_1$	$P2_1/c$	$P\overline{1}$
<i>a</i> , Å	7.275(2)	6.485(1)	6.649(1)	12.7395(8)	4.9604(7)
b, Å	8.003(2)	5.637(1)	5.9507(9)	6.6028(4)	11.506(2)
<i>c</i> , Å	9.359(3)	27.519(5)	15.626(3)	8.8693(6)	11.797(2)
α , deg.	90	90	90	90	91.482(2)
β , deg.	106.239(4)	90	90	102.643(1)	95.041(2)
γ, deg.	90	90	90	90	90.558(3)
$V, Å^3$	523.2(2)	1005.8(3)	618.2(2)	727.96(8)	670.4(2)
Z	4	8	4	4	4
Temperature	-60	-60	-60	-60	-60
λ	0.71073	0.71073	0.71073	0.71073	0.71073

Table 1. Crystal and refinement data for compounds 1–5

Density, g/cm^{-3}	2.513	2.614	2.277	2.973	3.209
μ , mm ⁻¹	5.988	6.229	5.075	8.551	9.284
θ range, deg	3.15-27.51	2.96 - 27.48	2.61-27.51	3.28-27.51	1.73-27.49
Limiting indices, h	-9 to 9	-8 to 8	-8 to 8	-15 to 16	-6 to 6
Limiting indices, k	-10 to 10	-7 to 6	-7 to 7	-6 to 8	-13 to 14
Limiting indices, l	-10 to 12	-34 to 35	-20 to 12	-11 to 10	-15 to 14
Reflections collected	3407	3315	1882	4880	4657
Independent reflections	1183	634	610	1666	2919
Parameters refined	67	47	43	69	109
GoF	1.073	1.197	1.076	1.194	1.049
$R1$, ^{<i>a</i>} w $R2^{b}$	0.025, 0.065	0.024, 0.061	0.022, 0.057	0.029, 0.083	0.043, 0.105
Largest shift/esd	0.001	-0.001	0.001	0.001	0.001
Largest peak, final	0.70	0.65	0.75	1.78	2.38
cvcle					

 ${}^{a}\text{R1} = \Sigma ||F_{o}| - |F_{c}||/\Sigma |F_{o}|.$ ${}^{b}\text{wR2} = [\Sigma[w(F_{o}^{2} - F_{c}^{2})^{2}]/\Sigma[w(F_{o}^{2})^{2}]]^{\frac{1}{2}}.$ $w = 1/[\sigma^{2}(F_{o}^{2}) + (aP)^{2} + (bP)], \text{ where } P = [max(F_{o}^{2} \text{ or } 0) + 2(F_{c}^{2})]/3.$

Table 2. Bond parameters (Å, deg.) for **1**

I(1)-C(3)	2.070(4)
O(1) - C(1)	1.310(4)
O(2)-C(1)	1.228(4)
C(1) - C(2)	1.477(5)
C(2)-C(3)	1.321(6)
I(1) - C(3) - C(2)	128.0(3)
C(1)-C(2)-C(3)	126.9(3)
O(1)-C(1)-O(2)	123.5(3)

across the carbon-carbon bond (Fig. 2). This assignment was subsequently confirmed by X-ray crystallography, and the thermal ellipsoid plot is presented in Fig. 1 (4). Bond distances and selected bond angles are gathered in Table 5. Although it is an unintended product and previously unknown molecule, it is probable that 3,3-diiodopropanoic acid could be deliberately prepared in reasonable yield by a slight modification of this method.

When the hydroiodination of propiolic acid is attempted at ambient temperature without reflux of the reaction mixture or addition of catalytic CuI, subsequent workup of the reaction by extraction yields small amounts of organic material, which, upon recrystallization, is clearly different from any of the other products. This material was identified by crystallography as *trans*-2,3-diiodoacrylic acid (**5** in Figs. 1 and 2), a product that undoubtedly results from reaction of propiolic acid with traces of I₂, which occur as a minor impurity in commercial HI solutions. The synthesis of this molecule by reaction of propiolic acid with I₂ has been previously reported,^[25–28] but none of these previous reports was accompanied by a crystallographic study. The bond distances and angles listed in Table 6 for **5** are for one of two independent (and disordered) molecules in the asymmetric unit.

As shown in Fig. 3, *trans*-2,3-diiodoacrylic acid is disordered in the crystalline state. The disorder is such that the positions of the two iodine atoms are fixed, and the remainder of the molecule is distributed between two highly

Table 3. Bond parameters (Å, deg.) for **2**

I(1)-C(3)	2.077(6)
O(1)-C(1)	1.218(7)
O(2)-C(1)	1.304(7)
C(1) - C(2)	1.489(7)
C(2)–C(3)	1.317(8)
I(1)-C(3)-C(2)	123.8(4)
C(1)-C(2)-C(3)	122.5(5)
O(1)-C(1)-O(2)	125.1(5)



Figure 2. Products resulting from the hydroiodination of propiolic acid.

overlapping and nearly coplanar orientations that are related to one another via a noncrystallographic twofold rotation axis. The position of this twofold axis may be easily visualized in Fig. 3 as the perpendicular bisector of the line segments joining atoms C(1A) with C(1B), C(2A) with C(2B), and C(3A) with C(3B). Because this twofold axis is noncrystallographic, that is, not required to exist by the symmetry of the crystal system, the site occupancy of these two orientations is not required to be 1:1. Refinement of the site occupancy as an optimal fit yielded a 7:3 occupancy ratio between the A and B orientations, respectively, where the A and B positions are those with atom labels bearing A or B in Fig. 3. A second molecule of trans-2,3-diiodoacrylic acid occurs in the asymmetric unit (not shown), is disordered in the same fashion, and was refined in the same manner as just described. Because of this disorder problem, anisotropic refinement was only attempted for the iodine atoms.

CONCLUSIONS

The foregoing results provide further, more complete characterization of the hydroiodination of propiolic acid and emphasize that the system is susceptible to a second hydroiodination in the presence of excess HI. To suppress this undesired further reaction, the synthesis of trans-3-iodoacrylic acid is best accomplished with a stoichiometric quantity of HI and elevated reaction temperatures

Table 4. I(1) - C(3)2.074(7)O(1) - C(1)1.324(9) O(2) - C(1)1.202(8)1.474(9)C(1) - C(2)C(2) - C(3)1.322(9)C(4) - O(1)1.47(1)I(1) - C(3) - C(2)123.8(5) C(1)-C(2)-C(3)119.4(5)

Bond parameters (Å, deg.) for 3

-	_
I(1)-C(3)	2.150(5)
I(2) - C(3)	2.147(4)
C(1)-C(2)	1.498(7)
C(2)-C(3)	1.520(7)
O(1)-C(1)	1.304(7)
O(2)-C(1)	1.224(7)
I(1) - C(3) - I(2)	110.5(2)
C(1)-C(2)-C(3)	112.0(4)

Table 5. Bond parameters (Å, deg.) for 4

(125–130°C). Because the reaction products (*cis*- and *trans*-iodoacrylic acid and 3,3-diiodopropanoic acid) crystallize in morphologically distinct forms (thick plates, needles, and blocks, respectively), these structural studies offer a convenient means for qualitative identification of the species isolated.

EXPERIMENTAL

Propiolic acid and HI (57% wt solution) were obtained from commercial sources and used as received. ¹H NMR spectra were obtained on a Varian XL-200AA spectrometer with chemical shifts referenced to the protonated solvent residual. The elemental analysis was performed by Canadian Microanalytical Service, Delta, British Columbia, Canada, upon a highly crystalline sample.

All crystals were coated with Paratone oil and mounted on a quartz fiber or nylon Cryoloop affixed to a goniometer head. All diffraction data were collected using a Bruker Smart 1000 CCD area-detector system using ω scans of 0.3 deg/ frame with 30, 60, or 90 second frames such that 1271 frames were collected for a full hemisphere of data. The first 50 frames were recollected at the end of the data collection to monitor for crystal decay, but no significant decomposition was observed. Cell parameters were determined using the program SMART.^[29] Data reduction and integration were performed with the software package SAINT,^[30] which corrects for Lorentz and polarization effects,

Table 6. Bond parameters (Å, deg.) for **5**

I(2)-C(3A)	2.01(2)
I(1)-C(2A)	2.11(1)
C(3A)-C(2A)	1.38(2)
C(2A)-C(1A)	1.48(2)
O(1A)-C(1A)	1.30(2)
O(2A)-C(1A)	1.24(2)
I(2) - C(3A) - C(2A)	131(1)
I(1)-C(2A)-C(3A)	119(1)



Figure 3. Illustration of the disorder in *trans*-2,3-diiodoacrylic acid about a noncrys-tallographic twofold axis. The iodine atoms are the only ones that are not disordered.

whereas absorption corrections were applied by using the program SADABS.^[30] Space groups for all the compounds structurally characterized by X-ray crystallography were initially determined with the aid of the program XPREP, which is part of the SHELX software package.^[31] In all structures, the positions of the iodine atom(s) were found via direct methods using the SHELX software, and the positions of the remaining nonhydrogen atoms were revealed by a subsequent cycle of least-squares refinement followed by difference Fourier synthesis. For both *cis*- and *trans*-3-iodoacrylic acid, the positions of the hydrogen atoms were visible in the final electron density maps and were refined. In the remaining structures, the hydrogen atoms were added in calculated positions and refined as riding atoms with isotropic displacement parameter values equal to 1.2 times those of the atoms to which they were attached. All refined structures were checked for higher symmetry using the crystallographic checking system provided by the International Union of Crystallography (http://journals.iucr.org/c/services/authorservices.html).

cis-3-Iodoacrylic Acid

cis-3-Iodoacrylic acid was synthesized via the method described by Abarbri et al.^[3] Diffraction-quality, thick colorless plate crystals were obtained by slow evaporation of an Et_2O solution.

trans-3-Iodoacrylic Acid and 3,3-Diiodopropanoic Acid

trans-3-Iodoacrylic acid was synthesized by the method described Zoller and Uguen^[4] starting from isolated *cis*-3-iodoacrylic acid. The crude product was

recrystallized from hexanes with a slight amount of CH₂Cl₂ added to slow crystallization. The white crystalline mass that formed proved to be ~1:1 mixture of *trans*-3-iodoacrylic acid (long, flat, white needle crystals) and 3,3-diiodopropanoic acid, **3** (large, white block crystals). The following data correspond to **3**: Mp (uncorrected) = 84.5–85 °C. ¹H NMR: δ (200 MHz; CDCl₃): 11.0 (1 H, s br, -CO₂H), 5.25 (1 H, t, *J* 7.3, -CHI₂), 3.81 (2 H, d, *J* 7.3, -CH₂). Anal. calcd. for C₃H₄I₂O₂: C, 11.06%; H, 1.24%. Found: C, 11.14%; H, 1.17%.

trans-3-Iodoacrylic Acid Methyl Ester

The methyl ester of *trans*-3-iodoacrylic acid was synthesized by the following method: *trans*-3-iodoacrylic acid (12.00 g, 61 mmol) was dissolved in 100 mL of distilled MeOH, and 0.5 mL of concentrated H₂SO₄ was added. This solution was vigorously refluxed for 24 h, cooled, and neutralized with aqueous NaHCO₃. The MeOH solvent was removed under reduced pressure, and the resulting reside was partitioned between 100 mL of H₂O and Et₂O. The Et₂O layer was separated, and the aqueous layer was extracted with 3 × 50-mL portions of Et₂O. The combined Et₂O extracts were filtered through anhydrous Na₂SO₄ and concentrated under reduced pressure to a volume of 25 mL. The remaining Et₂O was removed by distillation at ambient pressure, and the residual oil was crystallized upon cooling and standing. Pure *trans*-3-iodoacrylic acid methyl ester (2.56 g, 20% yield), the ¹H NMR spectrum of which agreed with literature data,^[24] was obtained. A diffraction-quality crystal was obtained directly from the cooled reaction melt.

trans-2,3-Diiodoacrylic Acid

Small quantities of *trans*-2,3-diiodoacrylic acid were obtained by treating propiolic acid with 57% aqueous HI at ambient temperature overnight. Workup of the reaction mixture as described for the preparation of *cis*-3-iodoacrylic acid^[2,4] yielded a small quantity of crude product that was mixture of elemental S₈ (resulting from extraction with aqueous thiosulfate) and *trans*-2,3-diiodoacrylic acid. Diffraction-quality white needle crystals of the latter were obtained by extracting the crude product with a minimal volume of Et₂O and slowly evaporating the solution to dryness.

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