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**Supporting information for article:**

# Diversity of *N*-triphenylacetyl-l-tyrosine solvates with halogenated solvents

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**Table S1** Experimental details

Experiments were carried out at 130 K with Cu *K*α radiation. H-atom parameters were constrained.

| | (I) | (II) | (III) |
|---|---|---|---|
| Crystal data | | | |
| Chemical formula | $C_{29}H_{25}NO_4{\cdot}CHCl_3$ | $C_{29}H_{25}NO_4{\cdot}CH_2Cl_2$ | $C_{29}H_{25}NO_4{\cdot}1.5(CHCl_3)$ |
| $M_r$ | 570.87 | 536.42 | 570.87 |
| Crystal system, space group | Monoclinic, $P2_1$ | Monoclinic, $P2_1$ | Orthorhombic, $P2_12_12$ |
| $a$, $b$, $c$ (Å) | 10.0732 (3), 10.0051 (2), 14.7474 (4) | 10.04475 (13), 9.82563 (12), 14.59465 (18) | 9.27898 (12), 32.3574 (4), 9.96336 (12) |
| α, β, γ (°) | 90, 109.945 (3), 90 | 90, 108.7422 (13), 90 | 90, 90, 90 |
| $V$ (Å$^3$) | 1397.14 (6) | 1364.05 (3) | 2991.44 (6) |
| $Z$ | 2 | 2 | 4 |
| $D_x$ (Mg m$^{-3}$) | 1.357 | 1.306 | 1.268 |
| μ (mm$^{-1}$) | 3.27 | 2.43 | 3.05 |
| Crystal size (mm) | 0.22 × 0.05 × 0.03 | 0.30 × 0.06 × 0.03 | 0.35 × 0.02 × 0.01 |
| Data collection | | | |
| Diffractometer | SuperNova, Single source at offset, Atlas | SuperNova, Single source at offset/far, Atlas | SuperNova, Single source at offset/far, Atlas |
| Absorption correction | Multi-scan | Gaussian | Multi-scan |
| $T_{min}$, $T_{max}$ | 0.408, 1.000 | 0.709, 1.000 | 0.713, 1.000 |
| No. of measured, independent and observed [$I > 2\sigma(I)$] reflections | 18782, 4905, 4842 | 20920, 5550, 5259 | 29945, 6241, 5987 |
| $R_{int}$ | 0.050 | 0.034 | 0.040 |
| $(\sin\theta/\lambda)_{max}$ (Å$^{-1}$) | 0.595 | 0.628 | 0.630 |
| Refinement | | | |
| $R[F^2 > 2\sigma(F^2)]$, $wR(F^2)$, $S$ | 0.048, 0.129, 1.03 | 0.035, 0.095, 1.05 | 0.032, 0.084, 1.01 |
| No. of reflections | 4905 | 5550 | 6241 |
| No. of parameters | 399 | 336 | 344 |

| | | | |
|---|---|---|---|
| No. of restraints | 35 | 1 | 0 |
| $\Delta\rho_{max}$, $\Delta\rho_{min}$ (e Å$^{-3}$) | 0.59, −0.41 | 0.42, −0.49 | 0.21, −0.27 |
| Absolute structure | Flack x determined using 2221 quotients [(I+)-(I-)]/[(I+)+(I-)] | Flack x determined using 2293 quotients [(I+)-(I-)]/[(I+)+(I-)] | Flack x determined using 2454 quotients [(I+)-(I-)]/[(I+)+(I-)] |
| Absolute structure parameter | 0.003 (12) | −0.009 (9) | 0.034 (6) |

| | (IV) | (V) |
|---|---|---|
| Crystal data | | |
| Chemical formula | $C_{29}H_{25}NO_4\cdot 0.1(CH_2Cl_2)$ | $C_{29}H_{25}NO_4\cdot 1.5(CHCl_3)$ |
| $M_r$ | 459.99 | 630.55 |
| Crystal system, space group | Orthorhombic, $P2_12_12_1$ | Monoclinic, $P2_1/c$ |
| $a$, $b$, $c$ (Å) | 9.28862 (9), 10.34013 (9), 24.3525 (2) | 10.00099 (16), 9.33129 (13), 31.8959 (5) |
| α, β, γ (°) | 90, 90, 90 | 90, 90.4958 (13), 90 |
| $V$ (Å$^3$) | 2338.95 (4) | 2976.48 (8) |
| $Z$ | 4 | 4 |
| $D_x$ (Mg m$^{-3}$) | 1.306 | 1.407 |
| μ (mm$^{-1}$) | 0.90 | 4.33 |
| Crystal size (mm) | 0.20 × 0.15 × 0.03 | 0.35 × 0.02 × 0.02 |
| Data collection | | |
| Diffractometer | SuperNova, Single source at offset/far, Atlas | SuperNova, Single source at offset/far, Atlas |
| Absorption correction | Multi-scan | Multi-scan |
| $T_{min}$, $T_{max}$ | 0.882, 1.000 | 0.773, 1.000 |
| No. of measured, independent and observed [$I > 2\sigma(I)$] reflections | 30278, 4276, 4225 | 20990, 6149, 4806 |
| $R_{int}$ | 0.024 | 0.046 |
| $(\sin\theta/\lambda)_{max}$ (Å$^{-1}$) | 0.602 | 0.630 |
| Refinement | | |
| $R[F^2 > 2\sigma(F^2)]$, $wR(F^2)$, $S$ | 0.031, 0.086, 1.05 | 0.058, 0.158, 1.04 |
| No. of reflections | 4276 | 6149 |
| No. of parameters | 494 | 372 |
| No. of restraints | 154 | 0 |

| | | |
|---|---|---|
| $\Delta\rho_{max}$, $\Delta\rho_{min}$ (e Å$^{-3}$) | 0.13, −0.14 | 0.84, −0.84 |
| Absolute structure | Flack x determined using 1770 quotients [(I+)-(I-)]/[(I+)+(I-)] | |
| Absolute structure parameter | 0.03 (3) | |

Computer programs: *CrysAlis PRO* 1.171.41.112a (Rigaku OD, 2021), SHELXT 2018/2 (Sheldrick, a2018), *SHELXL2018*/3 (Sheldrick, 2018).

**Table S2** Selected hydrogen-bond parameters

| *D*—H···*A* | *D*—H (Å) | H···*A* (Å) | *D*···*A* (Å) | *D*—H···*A* (°) |
|---|---|---|---|---|
| (I) | | | | |
| O1—H1···O3$^{ii}$ | 0.84 | 1.81 | 2.642 (4) | 173.7 |
| O4—H4···O2$^{iii}$ | 0.84 | 1.93 | 2.762 (4) | 174.3 |
| C8—H8···O3$^{i}$ | 0.95 | 2.53 | 3.427 (4) | 156.7 |
| C37$^{a}$—H37$^{a}$···O4 | 1.00 | 2.32 | 3.165 (7) | 141.8 |
| C26—H26···O3 | 0.95 | 2.27 | 2.946 (5) | 127.0 |
| (II) | | | | |
| O1—H1···O4$^{ii}$ | 0.84 | 1.80 | 2.641 (3) | 175.4 |
| O3—H3···O2$^{iii}$ | 0.84 | 1.93 | 2.772 (3) | 175.7 |
| C26—H26···O4 | 0.95 | 2.27 | 2.940 (3) | 126.6 |
| C6—H6···O2$^{iii}$ | 0.95 | 2.60 | 3.288 (3) | 129.2 |
| C6—H6···O4$^{i}$ | 0.95 | 2.53 | 3.409 (3) | 153.8 |
| (III) | | | | |
| O1—H1···O1$^{iv}$ | 0.84 | 1.83 | 2.635 (4) | 160.3 |
| O2—H2···O2$^{iv}$ | 0.84 | 1.80 | 2.627 (3) | 168.7 |
| O4—H4···O3$^{v}$ | 0.84 | 1.80 | 2.642 (3) | 175.9 |
| C36—H36···O3 | 0.95 | 2.32 | 2.825 (3) | 112.8 |
| C25—H25···O4$^{vi}$ | 0.95 | 2.41 | 3.292 (4) | 153.9 |
| (IV) | | | | |
| O4—H4···O3$^{vii}$ | 0.84 | 1.91 | 2.729 (2) | 166.0 |
| O1—H1···O4$^{iii}$ | 0.84 | 1.89 | 2.700 (2) | 161.4 |
| C16A$^{b}$—H16A$^{b}$···O3 | 0.95 | 2.04 | 2.708 (10) | 126.2 |
| (V) | | | | |

| | | | | |
|---|---|---|---|---|
| O1—H1···O2$^{viii}$ | 0.84 | 1.81 | 2.643 (3) | 174.6 |
| O4—H4···O3$^{iii}$ | 0.84 | 1.79 | 2.628 (3) | 173.0 |
| C22—H22···O3 | 0.95 | 2.38 | 2.822 (4) | 107.8 |

Symmetry code(s): (i) −*x*+2, *y*−1/2, −*z*+2; (ii) −*x*+2, *y*+1/2, −*z*+2; (iii) *x*, *y*−1, *z*; (iv) −*x*+2, −*y*+1, *z*; (v) *x*−1, *y*, *z*; (vi) *x*+1, *y*, *z*+1; (vii) *x*−1/2, −*y*+3/2, −*z*+1; (viii) −*x*+1, −*y*+2, −*z*+1.

**Table S3** Intermolecular hydrogen bonds in the known **TrCOTyr** polymorphs, solvates and cocrystals

| refcode | composition | 1 | 2 | 3 | 4 | 5 | 6 | 7 | 8 |
|---|---|---|---|---|---|---|---|---|---|
| EGUVUA | L-TrCOTyr | - | - | √ | - | - | √ | √ | 2D |
| EGUVUA01 | L-TrCOTyr | - | - | - | √ | √ | - | - | 1D |
| NOQSAQ | L-TrCOTyr·MeOH | √ | - | - | √ | - | √ | √ | 2D* |
| NOQSEU | L-TrCOTyr·EtOH | √ | - | - | √ | - | √ | √ | 2D* |
| NOQSIY | *rac*-TrCOTyr | - | - | - | √ | √ | - | - | 1D |
| NOQSOE | *rac*-TrCOTyr·MeOH | √ | - | - | √ | - | √ | √ | 2D* |
| NOQSUK | *rac*-TrCOTyr·EtOH | √ | - | - | √ | - | √ | √ | 2D* |
| NOQTAR | L-TrCOTyr·NPRD | - | - | - | - | - | √ | - | 2D** |
| NOQTEV | L-TrCOTyr·QX | - | - | - | - | - | √ | - | 1D |
| NOQTIZ | (L-TrCOTyr)$_2$·QX | - | - | - | √ | - | √ | - | 1D** |
| NOQTOF | (TrCOTyr)$_2$·BIPY | - | - | - | √ | - | - | - | 1D** |
| NOQTUL | (TrCOTyr)$_2$·BIPY | - | - | - | √ | - | - | - | 1D** |
| NOQVAT | L-TrCOTyr·DABCO | - | √ | - | √ | - | - | - | 1D |

1 – $R_2^2(8)$ carboxylic supramolecular synthon; 2 – O=CO-H···O=COH; 3 – O=CO-H···OH; 4 – O-H···O=COH; 5 – C=OO-H···O=CNH; 6 – O-H···O=CNH; 7 – O=CNH···O; 8 – dimensionality of the supramolecular structure by hydrogen bonds.

* alcohol molecules (methanol or ethanol) take part in the expansion of the supramolecular motif by O-H···O bonds

** diazaheterocyclic amine molecules interact with the **TrCOTyr** molecule through O-H···N hydrogen bonds and participate in the development of the supramolecular motif.