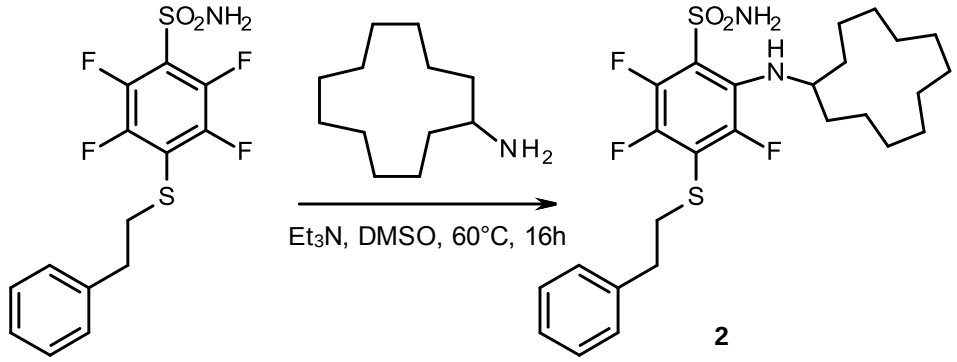
Supplementary Material

SUPPLEMENTARY MATERIALS AND METHODS

Chemical synthesis of the compounds

*The synthesis of 2-(Cyclododecylamino)-3,5,6-trifluoro-4-[(2-phenylethyl)thio]benzenesulfonamide (**2**).*



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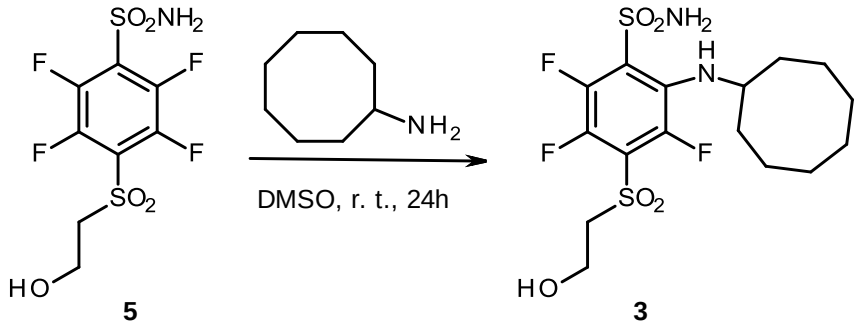
The mixture of 2,3,5,6-tetrafluoro-4-[(2-phenylethyl)thio]benzenesulfonamide (0.20 g, 0.55 mmol), Et_3N (0.08 mL, 0.57 mmol), DMSO (1 mL) and cyclododecylamine (0.15g, 0.57 mmol) was stirred at 60 ºC for 16 h. The mixture was then diluted with H_2O (20 mL) and extracted with EtOAc (3×10 mL). The combined organic phase was dried over $MgSO_4$ and evaporated under the reduced pressure. The product was purified by chromatography on a column of silica gel (0.040-0.063 mm) with EtOAc (5 %):$CHCl_3$, Rf = 0.65. Yield: 0.12 g, 41 %, mp 98-99 °C. ^{1}H NMR (300 MHz, $CDCl_3$): 1.34-1.75 (22H, m, cyclododecane), 2.94 (2H, t, J = 8.1 Hz, $SCH_2\underline{CH_2}$), 3.27 (2H, t, J = 8.1 Hz, $S\underline{CH_2}CH_2$), 3.84 (1H, br s, CH of cyclododecane), 5.40 (2H, s, SO_2NH_2), 7.20-7.36 (5H, m, ArH). ^{13}C NMR (75 MHz, $CDCl_3$): 21.3 (cyclododecane), 23.4 (cyclododecane), 23.5 (cyclododecane), 24.3 (cyclododecane), 24.6 (cyclododecane), 31.0 (cyclododecane), 35.4 ($S\underline{CH_2}CH_2$, t, J(^{19}F-^{13}C)= 4 Hz), 36.8 ($SCH_2\underline{CH_2}$), 53.6 (CH of cyclododecane, d, J (^{19}F-^{13}C) = 11 Hz), 116.7 (C1, dd, ^{1}J (^{19}F-^{13}C) = 12 Hz, ^{2}J (^{19}F-^{13}C) = 6 Hz), 119.8 (C4, t, J (^{19}F-^{13}C) = 18 Hz), 127.0 (Ar), 128.8 (Ar), 133.1 (C2, d, J (^{19}F-^{13}C) = 15 Hz), 139.4 (Ar), 141.7 (C5, ddd, ^{1}J (^{19}F-^{13}C) = 237 Hz, ^{2}J (^{19}F-^{13}C) = 16 Hz, ^{3}J (^{19}F-^{13}C) = 5 Hz), 145.1 (C6, ddd, ^{1}J (^{19}F-^{13}C) = 245 Hz, ^{2}J (^{19}F-^{13}C) = 12 Hz, ^{3}J(^{19}F-^{13}C)= 4 Hz), 148.4 (C3, d, J (^{19}F-^{13}C) = 243 Hz). ^{19}F NMR (282 MHz, $CDCl_3$): -120.1 (C3-F, d, J = 11 Hz), -138.6 (C5-F, dd, ^{1}J = 23 Hz, ^{2}J = 12 Hz), -144.7 (C6-F, d, J = 26 Hz). HRMS for $C_{26}H_{35}F_3N_2O_2S_2$ [$(M+H)^+$]: calc. 529.2165, found 529.2164.

*The synthesis of 3-(Cyclooctylamino)-2,5,6-trifluoro-4-[(2-hydroxyethyl)sulfonyl] benzenesulfonamide (**3**).*

The mixture of 2,3,5,6-tetrafluoro-4-[(2-hydroxyethyl)sulfonyl] benzenesulfonamide (**5**) (0.20 g, 0.59 mmol), DMSO (1 mL) and cyclooctylamine (0.15 g, 1.20 mmol) was stirred at ambient temperature for 24 h. The mixture was then diluted with H_2O (20 mL) and extracted with EtOAc (3×10 mL). The combined organic phase was dried over $MgSO_4$ and evaporated under the reduced pressure.



5 **3**

The product was purified by chromatography on a column of silica gel (0.040-0.063 mm) with EtOAc:$CHCl_3$ (1:1), Rf = 0.38. Yield: 0.10 g, 40 %, mp 89-90 °C. ^{1}H NMR (300 MHz, $CDCl_3$): 1.40-1.97 (14H, m, cyclooctane), 3.59 (2H, t, J = 5.7 Hz, $SO_2\underline{CH_2}CH_2$), 3.84-3.88 (1H, m, CH of cyclooctane), 4.11 (2H, t, J = 5.4 Hz, $SO_2CH_2\underline{CH_2}$), 6.08 (2H, s, SO_2NH_2), 6.74 (1H, br s, NH). ^{13}C NMR (75 MHz, $CDCl_3$): 23.5 (cyclooctane), 25.7 (cyclooctane), 27.4 (cyclooctane), 33.1 (cyclooctane), 56.3 (cyclooctane), 56.5 ($SO_2CH_2\underline{CH_2}$), 59.8 (cyclooctane), 60.8 ($SO_2\underline{CH_2}CH_2$), 115.8 (C4, dd, ^{1}J (^{19}F-^{13}C) = 13 Hz, ^{2}J (^{19}F-^{13}C) = 6 Hz), 126.5 (C1, t, J (^{19}F-^{13}C) = 16 Hz), 135.7 (C3, d, J (^{19}F-^{13}C) = 13 Hz), 137.0 (C6, dd, ^{1}J (^{19}F-^{13}C) = 246 Hz, ^{2}J (^{19}F-^{13}C) = 14 Hz), 144.7 (C2, d, J (^{19}F-^{13}C) = 253 Hz), 146.4 (C5, dd, ^{1}J (^{19}F-^{13}C) = 253 Hz, ^{2}J (^{19}F-^{13}C) = 16 Hz). ^{19}F NMR (282 MHz, $CDCl_3$): -125.9 (C2-F, s), -134.0 (C6-F, dd, ^{1}J = 25 Hz, ^{2}J = 12 Hz), -152.1 (C5-F, dd, ^{1}J = 24 Hz, ^{2}J = 4 Hz). HRMS for $C_{16}H_{23}F_3N_2O_5S_2$ [$(M+H)^+$]: calc. 445.1073, found 445.1077.

Protein preparation in *E. coli*

Preparation of human CA III

Complementary DNA (cDNA) of CA III was purchased from RZPD Deutsches Ressourcenzentrum für Genomforschung (Berlin, Germany). For recombinant protein expression, a nucleotide sequence encoding CA III from 4 to 260 amino acids was cut from full length cDNA with Van91I and Eco91I, blunt-ended with T4 DNA Polymerase and cloned via XhoI site into pET15b vector (Novagen) fusing a 6xHis-tag to the N terminus of the protein.

For protein expression, the plasmid pET-15b-6xHis-CA III was transformed into *E. coli* strain BL21(DE3). An overnight culture of plasmid-harboring cells was inoculated into fresh Luria-Bertani (LB) medium containing 60 μM $ZnCl_2$ and cultured at 37°C until an OD_{600} of 0.6-0.8 was reached. Expression of the target protein was induced by 0.2 mM isopropyl β-D-thiogalactoside (IPTG). The cells cultured at 30°C in the presence of 0.4 mM $ZnCl_2$ were harvested 4 h after induction and lysed by sonication. Soluble protein was purified using a Sepharose-IDA-Ni^{+2} affinity column, followed by anion exchange chromatography on CM-Sepharose (Amersham, Biosciences, Uppsala, NY).

Eluted protein was dialyzed into a storage buffer: 20 mM Mes, pH 6.2, 0.1 M NaCl and 2 mM DDT, sterile filtered and stored at -80°C. The purity of CA III preparations were analyzed by sodium dodecylsulfate-polyacrylamide gel electrophoresis (SDS-PAGE). Protein concentrations were determined by UV-VIS spectrophotometry using an extinction coefficient ε_{280} = 56045 M^{-1} cm^{-1} and confirmed by the standard Bradford method. Molecular mass of CA III was confirmed by mass spectrometry. The observed molecular mass was 31517.5 Da, theoretically predicted – 31648.6 Da. Theoretically predicted molecular mass without the methionine and water – 31517.4 Da, confirming the observed value.

Preparation of human CA VA

DNA fragment encoding full length CA VA was amplified by PCR from CA VA gene purchased from RZPD Deutsches Ressourcenzentrum für Genomforschung (Berlin, Germany), using forward primer with NdeI recognition site – 5' GGGCATATGTGTTCTCAGCGTTCCTG 3' and reverse primer with XhoI recognition site - 5' CTAATGCTCGAGGGACCTTGTGCCCTC 3' (recognition sites are underlined). PCR product was cloned into the bacterial expression vector pET21a (Novagen) via NdeI and XhoI restriction sites fusing a 6xHis-tag to the C termus of the protein.

Expression of recombinant CA VA was done in *E. coli* BL21(DE3) Codon Plus-RIL strain (Stratagene). Transformed cells colony was transferred to Luria-Bertani (LB) medium, containing 100 μg/ml ampicillin, 34 μg/ml chloramphenicol and grown at 37°C and 220 rpm for 16 h. Then the saturated culture was diluted (1:50) in fresh LB medium, containing 100 μg/ml ampicillin, 34 μg/ml chloramphenicol, 0.05 mM $ZnSO_4$ and grown to $OD_{600}\approx0.8$. The expression of recombinant CA VA was induced with 1 mM isopropyl β-D-thiogalactoside (IPTG) and 0.5 mM $ZnSO_4$. The culture was grown for 4 h at 30°C and 250 rpm. The cells were harvested by centrifugation at 4000g for 20 min at 4°C.

The pellet was suspended in lysis buffer (25 mM Tris, 1% Triton X-100, 0.1M NaCl, 2mM β-mercaptoethanol, 0.1M imidazole (*pH* 7.5) and 1 mM PMSF) containing protease inhibitor cocktail (Roche Applied Science, Indianapolis, IN). The cells were incubated at 4°C for 60 min and then disrupted by sonication. The supernatant, containing soluble proteins, was obtained after centrifugation at 20,000 × g for 25 min. Soluble protein was purified using a Sepharose-IDA-Ni^{+2} affinity column (GE Healthcare Bio-Sciences AB, Uppsala). Eluted protein was dialyzed against storage buffer (25 mM Tris, 0.1M NaCl, 1mM DTT, 10% glycerol *pH* 7.5) and stored at -80°C.

The purity of CA VA preparations were analyzed by sodium dodecylsulfate-polyacrylamide gel electrophoresis (SDS-PAGE). Protein concentrations were determined by UV-VIS spectrophotometry using an extinction coefficient ε_{280} = 71515 M^{-1} cm^{-1} and confirmed by the standard Bradford method.

Preparation of human CA VB

DNA encoding full length CA VB was purchased from RZPD Deutsches Ressourcenzentrum für Genomforschung (Berlin, Germany). CA VB from 40 to 317 amino acids was constructed using forward primer 5' CAAGCAACCCCCTAACTCGAGCATATC 3' and reverse primer 5' GATATGCTCGAGTTAGGGGGTTGCTTG 3' and cloned into the bacterial expression vector pET15b. CA VB was produced with the N-terminal 6xHis tag in *E. coli* Rosetta 2 (DE3) strain. CAVB was grown at 37°C in BHI medium containing 50 μg/ml ampicillin, 20 μg/ml chloramphenicol and 0.1 mM $ZnSO_4$ and induced with 0.1mM isopropylthio-β-galactoside (IPTG) and 0.1 mM $ZnSO_4$. After the induction, cells were cultured at 30 °C for 4 hours and 180 rpm. The cells were harvested by centrifugation at 4000g for 20 min at 4°C. Cell lysis was performed in 25mM Tris, 100 mM NaCl, 1mM phenylmethylsulfonyl fluoride, 0.5 % Triton X-100, pH 7.50, containing protease inhibitor cocktail (Roche applied Science, IN). The protein was purified using a Sepharose-IDA-Ni^{+2} affinity column (GE Healthcare Bio-Sciences AB, Uppsala), washed with Buffer A (25 mM Tris, 100 mM NaCl, 100 mM imidazole, 2% glycerol, 2 mM β – mercaptoethanol, pH 7.50). Bound protein was eluted with 500 mM imidazole in Buffer A. Molecular mass of CA VB was confirmed by mass spectrometry: observed molecular mass – 34062.2 Da, theoretically predicted – 34193.6 Da. The calculated molecular mass without methionine and water is 34062.6 Da.

Preparation of human CA XIV

DNA fragment encoding CA XIV from 20 to 280 amino acids was amplified by PCR from full CA XIV gene purchased from RZPD Deutsches Ressourcenzentrum für Genomforschung (Berlin, Germany), using forward primer with NdeI recognition site – 5' CAGATCATATGCAACACTGGACGTAT 3' and reverse primer with XhoI recognition site - 5' ACGACTCGAGTTATTGGATGAAAGAAGCAAA 3' (recognition sites are underlined). PCR product was cloned into the bacterial expression vector pET15b (Novagen) via NdeI and XhoI restriction sites fusing a 6xHis-tag to the N terminus of the protein.

The recombinant CA XIV has catalytic domain and lacks signal and transmembrane domains. Expression of recombinant CA XIV was done in *E. coli* Rosetta2 (DE3) strain (Novagen). Transformed cells colony was transferred to LB medium, containing 100 μg/ml ampicillin, 34 μg/ml chloramphenicol and grown at 37°C and 220 rpm for 16 h. Then the saturated culture was diluted (1:50) in fresh LB medium, containing 50 μg/ml ampicillin, 20 μg/ml chloramphenicol, 0.05 mM $ZnSO_4$ and grown to $OD_{600}\approx0.8$. The expression of recombinant CA XIV was induced with 0.4 mM isopropyl β-D-thiogalactoside (IPTG) and 0.5 mM $ZnSO_4$. The culture was grown over night at 16°C and 250 rpm. The cells were harvested by centrifugation at 4000g for 20 min at 4°C.

The pellet was suspended in lysis buffer (25 mM Tris, 1% Triton X-100, 0.1 M NaCl, 0.05 M imidazole (*pH* 8) and 1 mM PMSF) containing protease inhibitor cocktail (Roche applied Science, Indianapolis, IN). The cells were incubated at 4°C for 60 min and then disrupted by sonication. The supernatant, containing soluble proteins, was obtained after centrifugation at 20,000 × g for 25 min. Soluble protein was purified using an affinity column (Ni^{+2} Chelating Sepharose, FF GE Healthcare Bio-Sciences, Uppsala, Sweden), followed by a CA-affinity column containing p-aminomethylbenzene sulfonamide-agarose (Sigma-Life Science Aldrich). Eluted protein was dialysed against storage buffer (25 mM Tris, 0.1 M NaCl, *pH* 8) and stored at -80°C.

The purity of CA XIV preparations were analyzed by sodium dodecylsulfate-polyacrylamide gel electrophoresis (SDS-PAGE). Protein concentrations were determined by UV-VIS spectrophotometry using an extinction coefficient ε_{280} = 41432.5 M^{-1} cm^{-1} and confirmed by the standard Bradford method. Molecular mass of CA XIV was confirmed by mass spectrometry: observed molecular mass – 31696.9 Da, theoretically predicted – 31698.2 Da. Theoretically predicted molecular mass of CA XIV without cysteine bridge – 31697.2.

Preparation of chimeric chCA XII

The expression vector pET15b-CA II, encoding full length CA II (1-260) [42] was used in site-directed mutagenesis. In CA II active site located residues A65, N67, I91, F130, V134, and L203 were replaced to S, K, T, A, and S, N respectively. For each mutagenesis reaction two oligonucleotide primers (sense and antisense) with desired mutation were used: **A65S_N67K_s**: CCT CAA CAA TGG TCA TAG TTT CAA GGT GGA GTT TGA TGA C and **A65S_N67K_a**: GTC ATC AAA CTC CAC CTT GAA ACT ATG ACC ATT GTT GAG G; **I91T_s**: GGC ACT TAC AGA TTG ACT CAG TTT CAC TTT C and **I91T_a**: GAA AGT GAA ACT GAG TCA ATC TGT AAG TGC C; **F130A_s**: ACC AAA TAT GGG GAT GCT GGG AAA GCT TCG CA and **F130A_a**: TGC GAA GCT TTC CCA GCA TCC CCA TAT TTG GT; **V134S_s**: GGA TTT TGG GAA AGC TTC GCA GCA ACC TGA TG and **V134S _a**: CAT CAG GTT GCT GCG AAG CTT TCC CAA AAT CC; **L203N_s**: GAC CAC CCC TCC TCT TAA TGA ATG TGT GAC CTG G and **L203N_a**: CCA GGT CAC ACA TTC ATT AAG AGG AGG GGT GGT C. PCR was carried out with high fidelity *Pfu* DNA polymerase (Thermo Fisher Scientific). Composition of

PCR: 1x polymerase buffer (with $MgSO_4$), 50 ng template DNA, 0.2 mM dNTPmix, 125 ng each sense and antisense primer, and 1.5 u *Pfu* DNA polymerase (native). Thermal cycling conditions: initial denaturation - 95°C for 10 min, then 18 cycles: 95°C for 3 min, annealing - 71°C (**A65S_N67K**), 69°C (**I91T, F130A**), 69°C (**V134S**), 71°C (**L203N**) for 2 min, extension - 72°C for 8 min, final extension – one time 72°C for 10 min. After temperature cycling, PCR product was treated with 10 u *Dpn I* restriction endonuclease in order to digest the parental DNA template and to select new synthesized mutated DNA (site-directed mutagenesis was carried out according to [79] and the instruction manual by Stratagene. The mutations were confirmed by DNA sequencing.

Expression of the chimeric CA XII protein was done in *E. coli* BL21(DE3) strain. Transformed cells colony was transferred to LB medium, containing 100 μg/ml ampicillin, was grown at 37°C, 220 rpm for 16 h. Then the saturated culture was diluted (1:50) in fresh LB medium, containing 100 μg/ml ampicillin and 60 μM $ZnSO_4$ and grown to $OD_{600} \approx 0.8$. The expression of chimeric CA XII was induced with 0.2 mM isopropyl-β-D-thiogalactoside (IPTG) and 0.4 mM $ZnSO_4$. The cells were grown over night at 19°C and 220 rpm and harvested by centrifugation at 4000g for 20 min. at 4°C.

The collected biomass was suspended in lysis buffer (20 mM Hepes, and 0.15 M NaCl, pH 6.7), incubated at 4°C for 60 min and then disrupted by sonication. Debris of cells and unsoluble proteins were precipitate by centrifugation at 30,000g for 25 min. The soluble chimeric CA XII protein was purified using a metal chelate and CA-affinity chromatography. For the metal chelate chromatography, the column was equilibrated with 20 mM Hepes, 0.15 M NaCl (pH 6.7). For elution of chimeric CA XII 20 mM Hepes, 0.15 M NaCl (pH 6.7) and 0.2 M imidazole (pH 6.7) solution was used. Eluted protein was purified using a CA-affinity column containing p-aminomethylbenzene sulfonamide-agarose (Sigma-Life Science Aldrich). Sorbent was equilibrated with 20 mM Hepes, 0.15 M NaCl (pH 7.8). For the protein elution 0.1 M sodium acetate and 0.5 M sodium perchlorate, (pH 5.6) was used. Eluted chimeric protein was dialyzed into storage buffer containing 20 mM Hepes, 0.05 M NaCl, pH 7.9, and stored at -80°C.

The purity of the chimeric CA XII preparations was analyzed by SDS-PAGE. Protein concentrations were determinated by UV-vis spectrophotometry using extinction coefficient ε_{280} = 50420 M^{-1} cm^{-1} and confirmed by standard Bradford method.

Molecular mass of chimera CA XII was confirmed by mass spectrometer: observed molecular mass – 29047.1 Da, theoretically predicted – 29176.8 Da. Theoretically predicted molecular mass without methionine and water – 29045.8 Da.

Preparation of chimeric chCA IX

The expression vector pET15b-CA II, encoding full length CA II (1-260)[42] was used in site-directed mutagenesis. In CA II active site located residues A65, N67, I91, F130, V134, and L204 were replaced to S, Q, L, V, and L, A respecively. For each mutagenesis reaction two oligonucleotide primers (sense and antisense) with desired mutation were used: **A65S_s**: CTC AAC AAT GGT CAT AGT TTC AAC GTG GAG and **A65S_a**: CTC CAC GTT GAA ACT ATG ACC ATT GTT GAG; **N67Q_s**: TGG TCA TAG TTT CCA GGT GGA GTT TGA TGA C and **N67Q_a**: GTC ATC AAA CTC CAC CTG GAA ACT ATG ACC A; **I91L_s**: CAC TTA CAG ATT GCT TCA GTT TCA CTT TCA CTG and **I91L_a**: CAG TGA AAG TGA AAC TGA AGC AAT CTG TAA GTG; **F130V_V134L_s**: CCA AAT ATG GGG ATG TTG GGA AAG CTT TGC AGC AAC CTG and **F130V_V134L _a**: CAG GTT GCT GCA AAG CTT TCC CAA CAT CCC CAT ATT TGG;**L203A_s**: CCA CCC CTC CTC TTG CGG AAT GTG TGA CC and **L203A_a**: GGT CAC ACA TTC CGC AAG AGG AGG GGT GG. PCR was carried out with high fidelity *Pfu* DNA

polymerase (Thermo Fisher Scientific). Composition of PCR: 1x polymerase buffer (without $MgSO_4$), 4 mM $MgSO_4$, 50 ng template DNA, 0.2 mM dNTPmix, 125 ng each sense and antisense primer, and 1.5 u *Pfu* DNA polymerase (native). Thermal cycling conditions: initial denaturation - 95°C for 10 min, then 18 cycles: 95°C for 3 min, annealing - 69°C (N67Q and L203A), 70°C (I91L and F130V_V134L), 71°C (A65S), for 2 min, extension - 72°C for 8 min, final extension – one time 72°C for 10 min. After temperature cycling, PCR product was treated with 10 u *Dpn I* restriction endonuclease in order to digest the parental DNA template and to select new synthesized mutated DNA (site-directed mutagenesis was carried out according to [79] and the instruction manual by Stratagene). The mutations were confirmed by DNA sequencing.

Expression of the chimeric CA IX protein was done in *E. coli* BL21(DE3) strain. Transformed cells colony was transferred to LB medium, containing 100 μg/ml ampicillin, grown at 37°C and 220 rpm for 16 h. Then the saturated culture was diluted (1:50) in fresh LB medium, containing 100 μg/ml ampicillin and 60 μM $ZnSO_4$ and grown to $OD_{600} \approx 0.8$. The expression of chimeric CA IX was induced with 0.2 mM isopropyl-β-D-thiogalactoside (IPTG) and 0.4 mM $ZnSO_4$. The cells were grown over night at 19°C and 220 rpm, harvested by centrifugation at 4000g for 20 min. at 4°C.

The collected biomass was suspended in lysis buffer (20 mM Hepes, and 0.15 M NaCl, pH 7.8), incubated at 4°C for 60 min and then disrupted by sonication. Debris of cells and unsoluble proteins were precipitate by centrifugation at 30,000g for 25 min. The soluble chimeric CA IX protein was purified using a metal chelate and CA-affinity chromatography. For the metal chelate chromatography, the column was equilibrated with 20 mM Hepes, 0.15 M NaCl (pH 6.7). For elution of chimeric CA IX 20 mM Hepes, 0.15 M NaCl (pH 6.7) and 0.2 M imidazole (pH 6.7) solution was used. Eluted protein was purified using a CA-affinity column containing p-aminomethylbenzene sulfonamide-agarose (Sigma-Life Science Aldrich). Sorbent was equilibrated with 20 mM Hepes, 0.15 M NaCl (pH 7.8). For the protein elution 0.1 M sodium acetate and 0.5 M sodium perchlorate, (pH 5.6) was used. Eluted chimeric protein was dialyzed into storage buffer containing 20 mM Hepes, 0.05 M NaCl, pH 7.9, and stored at -80°C.

The purity of the chimeric CA IX preparations was analyzed by SDS-PAGE. Protein concentrations were determined by UV-vis spectrophotometry using extinction coefficient $\varepsilon_{280} = 50420\ M^{-1}\ cm^{-1}$ and confirmed by standard Bradford method.

Molecular mass of chimera CA IX was confirmed by mass spectrometer: observed molecular mass – 29069.3 Da, theoretically predicted – 29199.9 Da. Theoretically predicted molecular mass without methionine and water – 29068.7 Da.

Crystallization

Supplementary Table 1. Crystallization buffers, space groups, and the cell parameters of CA crystals. The PDB IDs are listed in Table 2 of the manuscript.

Crystal structure	Crystallization buffer	Space group and cell parameters
hCA2-**VD12-09**	0.1 M sodium bicine (pH 9.0), 0.2 M ammonium sulfate and 2M sodium malonate (pH 7.0)	$P2_1$; a=*42.02*, b=*40.90*, c=*71.60*, $\alpha=\gamma=90.00^\circ$, $\beta=104.04^\circ$;
chCA12-**VD12-09**	90% of 0.1 M sodium bicine (pH 9.0), 0.2 M ammonium sulfate and 2M sodium malonate (pH 7.0)	$P2_1$; a=*41.82*, b=*41.06*, c=*71.47*, $\alpha=\gamma=90.00^\circ$, $\beta=103.56^\circ$;
chCA9-**VD12-09**	0.1 M sodium bicine (pH 9.0), 0.2 M ammonium sulfate and 2 M sodium malonate (pH 7.0)	$P2_1$; a=*42.02*, b=*41.34*, c=*72.19*, $\alpha=\gamma=90.00^\circ$, $\beta=104.14^\circ$;
chCA12-**VD11-4-2**	90% of 0.1 M sodium bicine (pH 9.0), 0.2 M ammonium sulfate and 2 M sodium malonate (pH 7.0)	$P2_1$; a=*41.84*, b=*41.01*, c=*72.01*, $\alpha=\gamma=90.00^\circ$, $\beta=103.56^\circ$;
hCA2-**VD11-4-2**	0.1 M sodium bicine (pH 9.0), 0.2 M ammonium sulfate and 2 M sodium malonate (pH 7.0)	$P2_1$; a=*42.24*, b=*41.52*, c=*71.94*, $\alpha=\gamma=90.00^\circ$, $\beta=104.42^\circ$;
chCA9-**VD11-4-2**	95% of 0.1 M sodium bicine (pH 9.0), 0.2 M ammonium sulfate and 2 M sodium malonate (pH 7.0)	$P2_1$; a=*41.93*, b=*41.21*, c=*72.07*, $\alpha=\gamma=90.00^\circ$, $\beta=104.21^\circ$;
hCA12-**VD11-4-2**	0.1 M ammonium citrate (pH 5.0) and 18% PEG4000	P1; a=*46.52*, b=*75.21*, c=*77.89*, $\alpha=109.42^\circ$, $\beta=101.60^\circ$, $\gamma=107.95^\circ$;
hCA2-**VD10-35**	0.1 M sodium bicine (pH 9.0), 0.2 M ammonium sulfate and 2 M sodium malonate (pH 7.0)	$P2_1$; a=*42.10*, b=*40.97*, c=*71.69*, $\alpha=\gamma=90.00^\circ$, $\beta=104.14^\circ$;
4HU1: hCA2-**VD10-35**	0.1 M sodium citrate (pH 5.5), 0.1M sodium acetate (pH 4.5), and 26 % PEG4000	$P2_12_12_1$; a=*56.27*, b=*57.39*, c= *159.55*, $\alpha=\beta=\gamma=90^\circ$